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Synthesiology

English edition

Synthesiology editorial board

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MESSAGES FROM THE EDITORIAL BOARD

There has been a wide gap between science and society. The last three hundred years of the history of modern science indicates to us that many research results disappeared or took a long time to become useful to society. Due to the difficulties of bridging this gap, it has been recently called the valley of death or the nightmare stage (Note 1). Rather than passively waiting, therefore, researchers and engineers who understand the potential of the research should be active.

To bridge the gap, technology integration (i.e. *Type 2 Basic Research* – Note 2) of scientific findings for utilizing them in society, in addition to analytical research, has been one of the wheels of progress (i.e. *Full Research* – Note 3). Traditional journals, have been collecting much analytical type knowledge that is factual knowledge and establishing many scientific disciplines (i.e. *Type 1 Basic Research* – Note 4). Technology integration research activities, on the other hand, have been kept as personal know-how. They have not been formalized as universal knowledge of what ought to be done.

As there must be common theories, principles, and practices in the methodologies of technology integration, we regard it as basic research. This is the reason why we have decided to publish "*Synthesiology*", a new academic journal. *Synthesiology* is a coined word combining "synthesis" and "ology". Synthesis which has its origin in Greek means integration. Ology is a suffix attached to scientific disciplines.

Each paper in this journal will present scenarios selected for their societal value, identify elemental knowledge and/or technologies to be integrated, and describe the procedures and processes to achieve this goal. Through the publishing of papers in this journal, researchers and engineers can enhance the transformation of scientific outputs into the societal prosperity and make technical contributions to sustainable development. Efforts such as this will serve to increase the significance of research activities to society.

We look forward to your active contributions of papers on technology integration to the journal.

Addendum to Synthesiology-English edition,

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Note 1 : The period was named "nightmare stage" by Hiroyuki Yoshikawa, President of AIST, and historical scientist Joseph Hatvany. The "valley of death" was by Vernon Ehlers in 1998 when he was Vice Chairman of US Congress, Science and Technology Committee. Lewis Branscomb, Professor emeritus of Harvard University, called this gap as "Darwinian sea" where natural selection takes place.
Note 2 : *Type 2 Basic Research*
This is a research type where various known and new knowledge is combined and integrated in order to achieve the specific goal that has social value. It also includes research activities that develop common theories or principles in technology integration.
Note 3 : *Full Research*
This is a type of research where the theme is placed within a scenario of future society, and where a framework is developed in which researchers from a wide range of research fields can participate in studying actual issues. This research is done continuously and concurrently from *Type 1 Basic Research* (Note 3) to *Product Realization Research* (Note 5), centered by *Type 2 Basic Research* (Note 4).
Note 4 : *Type 1 Basic Research*
This is an analytical research type where unknown phenomena are analyzed, by observation, experimentation, and theoretical calculation, to establish universal principles and theories.
Note 5 : *Product Realization Research*
This is a type of research where the results and knowledge from *Type 1 Basic Research* and *Type 2 Basic Research* are applied to embody the use of a new technology in society.

Synthesiology – English edition Vol.1 No.4

Contents

Development of high power and high capacity lithium secondary battery based on the advanced nanotechnology

— The convergence innovation strategy employing the inter-disciplinary research and inter-organization straightforward technology transfer —

Itaru Honma

[Translation from *Synthesiology*, Vol.1, No.4, p.247-258 (2008)]

The development of high power and high capacity lithium secondary battery has been executed in a new strategic innovation scheme. This paper reports the significance of adopting the idea of interdisciplinary research to straightforward technology transfer from university, through AIST and battery company down to automobile company. A joint project of these organizations covering the upper stream science (at a university) and the down stream battery products (at a company) has been found a good scheme for rapid R&D when the university or AIST has innovative seeds. AIST has superiority in producing new technological seeds because of the interdisciplinary research between energy and nanotechnology as well as a wide spectrum of research fields covered by the institute. In this paper, an application of nanocrystalline electrode materials to high power battery is reported where the technology was successfully transferred to the commercial development of superior battery products.

Keywords : Lithium secondary battery, nanocrystal, high capacity and high power electrode, plug-in hybrid car, hybrid electric vehicle (HEV)

1 Background of research

Japan and other advanced nations are faced with two major energy crises today. One is structural energy limitation due to rapid increase of energy demand from BRIC countries and fierce international competition over procurement of resource for third world economic growth. Second is decrease in food production due to dramatic changes in atmospheric environment caused by global warming, as well as increased risk to sustainable biosphere due to reduction of space suitable for human survival, caused by increased abnormal climate. Japan, which is a resource-poor country, suffers from uncertainty factor of energy supply that is the foundation of nation's economic development, and innovations in energy technology is the most important issue for ensuring national energy security.

High-power lithium secondary battery is highly effective against global warming, and it is expected to accelerate the market entry of plug-in hybrid electric vehicles (HEV) and electric vehicles (EV) that may raise industrial competitiveness. Fierce competition is taking place throughout the world for its development. The desired high output performance of battery cannot be achieved with conventional material technology, and innovations in battery technology have been awaited. With the background of recent advances in nanoscience and nanotechnology, there is great interest in development of high-power high-capacity electrode material based on fine nanostructure control of electrochemical active materials.

The Author became interested in innovative active materials based on state-of-art nanotechnology, and worked on the development of high-speed charge-discharge electrode. Since charge transfer rate increases dramatically in nanocrystal where the diffusion length of ions becomes extremely short, the possibility of designing high-speed charge-discharge battery has been theorized. The Author's research team applied advanced nanotechnology process and fabricated electrodes with nanocrystal and nanoporous structures that could not be made with conventional material science, and showed that high output property that far surpassed conventional performance could be achieved under laboratory conditions. Therefore, we decided to investigate whether these new material design concepts could be used as power source of next-generation HEV in industry-academia-government collaboration project, with involvement of battery and automobile manufacturers. In this paper, the efficacy of nanotechnology for innovations in the field of energy storage will be discussed, and the efficacy of vertical collaboration project of industry-academia-government for swift commercialization of innovation will be addressed, as well as introducing the actual results of the technological development.

2 Development of innovative battery technology through fusion of nanotechnology and energy technology

Figure 1 is the Ragone plot of performances of lithium secondary batteries that have been realized dramatically in recent years, and examples of innovative energy devices and products. Currently they are put to practical use in cell phones and laptop computers. These devices use small low-capacity lithium battery and do not require high output density. Recently, middle-sizing and higher power for lithium battery became possible, and the batteries are used as power source for electric bicycles and power tools. The frontline

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of lithium battery technology is moving from small to large devices, and higher power, higher capacity, and lower cost are important issues assuming application as power source for automobiles. As shown in Fig. 1, the frontline of battery technology application is shifting from lower right to upper left. The middle- and large-size, highly reliable lithium battery is expected to become central energy technology for robots, HEVs, and recyclable energy in the future. To accomplish this, it is necessary to develop high-power cell of several kW/kg level as mentioned above, and innovative technological development using nanotechnology is drawing great expectation and investments.

In fact, assuming use as auxiliary power for clean vehicles represented by hybrid and fuel cell cars that are key technologies to counter global warming, power source with both sufficient energy and output densities is necessary. Figure 2 is a Ragone plot of target performance values of auxiliary power source for HEV of Japanese automobile manufacturers and U.S. Department of Energy (DOE). When targeting energy regeneration for general-use automobile, about 30 Wh/kg energy density and 3 kW/kg output density are required for battery cell, and such performance is intermediate level for lithium secondary battery and electric double-layer capacitor (EDLC). This performance (30 Wh/kg, 3 kW/kg) is equivalent to charge-discharge rate of 100 times per hour (completely charged in only 36 sec), and it is impossible to construct a storage device with such energy and output densities using bulk size intercalation electrode materials currently available. If ion intercalation mechanism was used for secondary battery active material, charge-transfer rate must be improved 100 times. That is, if storage mechanism that is midway between secondary battery and EDLC is used to obtain both energy and output densities, dramatic acceleration of ion dispersal within electrode and electronic conductivity must be achieved simultaneously.

This paper is a report of the result of the industry-academia-government vertical collaboration project where the goal was development of nanoporous electrode composed of nanospace

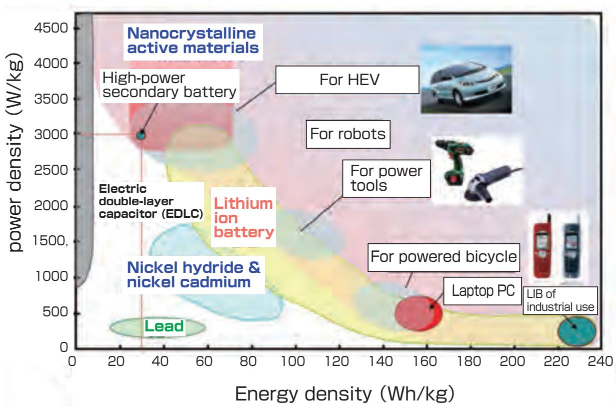

Fig. 1 Industrial products realized by innovations in battery technology.

and nanocrystal active material, to create power source that enables high-speed input/output of electric energy, and to create high-power battery that can charge and discharge 100 times faster compared to ordinary lithium secondary battery. Particularly, I shall explain the original concept of the National Institute of Advanced Industrial Science and Technology (AIST) that conducted application of nanocrystal electrode material to high-power battery, and describe how nanotechnology can greatly contribute to the innovation of electric power storage. In the vertical collaboration R&D, automobile manufacturer who was end user participated from the planning stage of the project. For electrode material development that enabled high-output power source performance (strategic goal value in Fig. 2 about 3 kW/kg) required for plug-in HEV, we had the automaker indicate the direction of basic research at university and AIST, to maintain sufficient market competitiveness in terms of safety and low cost. To demonstrate the efficacy of vertical collaboration R&D that lead to product realization in shortest distance without major changes in specification due to result of core technology development, we conducted industry-academia-government collaboration product by four organizations with support from New Energy and Industrial Technology Development Organization (NEDO).

First, I shall explain the physicochemical basis of how high-power battery that is an innovation of storage technology using nanotechnology can be realized. The storage mechanism originates from electrochemical reaction accompanied by charge transfer by ion diffusion and electronic conduction. Here, diffusion length L in certain time can be expressed by the following equation, when diffusion coefficient of lithium ion in solid is D_{Li} and diffusion time is τ.

$$L = (D_{Li}\,\tau)^{1/2} \qquad (1)$$

If the diffusion coefficient of lithium ion in active material is estimated to be about 10^{-13} cm^2/s, time τ required for ion

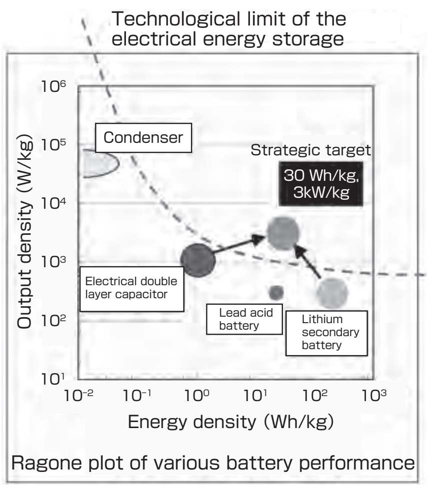

Fig. 2 Frontier in battery performance.

to diffuse inside the active material of width 5 nm is 1 sec at most. When the diffusion coefficient of ion in electrolyte within the pore is estimated to be about 10^{-6} cm^2/s, the time required for diffusion inside pore with length 10 μm can be estimated as 1 sec at most. Therefore, as shown in Fig. 3, if fast ion diffusion (k_1) in electrolyte within nanopore and ion diffusion rate (k_3) in solid at nanometer level within active material are used, charge and discharge can be realized in second order even if the particle size of nanoporous structure electrode is larger than micrometer. However, this is theoretical consideration under condition that electronic conduction (k_4) of active material framework and surface reaction of active material (k_2) are sufficiently fast in the rate determining process shown in Fig. 3. On the other hand, since specific surface area is large in nanoporous electrode, there is interest in energy property unique to nano material such as lithium storage caused by electrochemical reaction on the surface. If the surface is utilized, there may be possibility of storing lithium above stoichiometric composition.

Various nano-size electrode active materials were made based on migration kinetics at AIST to check the high capacity electrode property of nanoporous crystal material, and high-speed charge-discharge property was evaluated. In principle, if nanoporous electrode could be fabricated, charge-discharge in 36 sec, which was required as power source of HEV, would be possible. To accomplish this, it was necessary to integrate the elemental technologies of the frontier of nanotechnology fields such as solution process, molecular template synthesis technology, self-assembling process, and mass synthesis process for nanocrystals, and then to check the efficacy of innovative electrode active materials at battery cell level jointly with manufacturers. The competition for development is becoming fierce around the world since such high-power battery is long awaited in the industry and the market scale is great.

3 Execution and result of R&D

I shall describe the outline of development of high-power lithium secondary battery through interdisciplinary fusion and industry-academia-government vertical collaboration conducted as NEDO project, and discuss whether the strategy for "shortening distance" to innovation was effective. This R&D was a vertical collaboration project by four organizations, Nagasaki University, AIST, Hitachi Maxell, Ltd., and Fuji Heavy Industries Ltd., as "Research and Development of High Capacity Secondary Battery by Low Resistance, High Ion Diffusion Nanoporous electrode" under the R&D for Practical Utilization of Nanotechnology and Advanced Materials conducted in FY 2005~2007. This project was conducted to develop high-power lithium secondary battery for HEV using advanced nanotechnology, and it was original because it used nanotech in energy technology effectively. It was characteristic that the end user automobile manufacturer was included in the project from commencement, and it was also distinct that the use of electrode technology of university and AIST as power source of HEV in short period was placed as central topic of R&D.

Nagasaki University, which was located upstream, investigated the inorganic chemical synthesis process of nanoporous electrode material from which high output property could be expected, from standpoint of basic chemistry, and developed new synthesis method that could be applied to practical electrode. Mesoporous material using molecular template such as surfactants could be applied to amorphous structure such as silica but were difficult to apply to crystalline active material such as $LiCoO_2$ and $LiFePO_4$ that were positive electrode materials for lithium secondary battery. Therefore, a process to fabricate inverse-opal form electrode framework using template structure with colloid polystyrene (PS) was developed, as shown in Fig. 4.

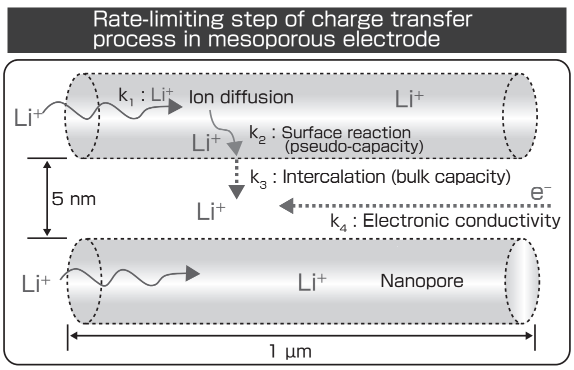

Fig. 3 Charge transfer process and rate-controlling step within electrode.

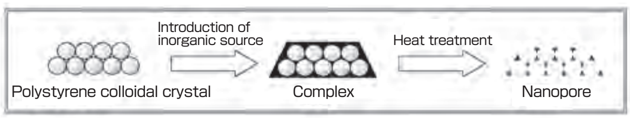

Fig. 4 Potential of university: nanoporous electrode synthesis process.

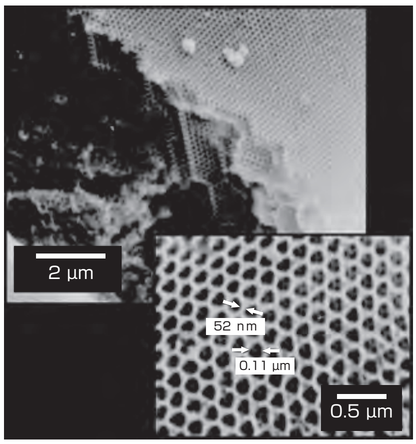

Fig. 5 Titania nanoporous electrode.

The example of synthesis of titania nanoporous electrode is shown as specific example. Ethanol solution of titanium alkoxide was added to PS colloid crystal, and the solution was mixed with titania nanoporous body consisting of anatase crystal by firing at 450 ºC, and followed by niobium alkoxide, to obtain $Ti_xNb_{1-x}O_2$ nanoporous electrode (Fig. 5). Because these electrodes had sequential structure of three-dimensional framework and had regular arrangement of nanopores and active materials, it was possible to obtain high ion conductivity, electronic conductivity, and lithium storage capacity.

Next, AIST conducted synthesis and structural evaluation of high-power battery electrode material using these nanocrystals followed by electrochemical property evaluation, and investigated the physical chemical mechanism of high-speed charge transfer property for how such innovative energy property could be obtained from this nanomaterial technology[1].

In actual material development, the electrochemically active nanocrystal titania was set as model material for high-power electrode, and anatase and rutile structures were prepared in size ranges 6 nm ~ 100 nm, to conduct systematic evaluation of size dependency of lithium battery electrode property[2][3].

In nanocrystal titania, specific surface area was large since the particle diameter was in nanosize range, and surface energy storage property was readily available, and surface and interior of solid were thought to be two phases in coexistence as region that reacted electrochemically with lithium. That is, there were two different lithium storage mechanisms, where former was surface lithium adsorbed accompanied by charge transfer on surface of nanocrystal, while latter was lithium stored by intercalation inside the nanocrystal. The faradaic capacity by surface adsorption manifested as pseudo-capacity, and stored lithium inside the solid manifested as intercalation capacity. These two different lithium storage mechanisms were shown clearly in

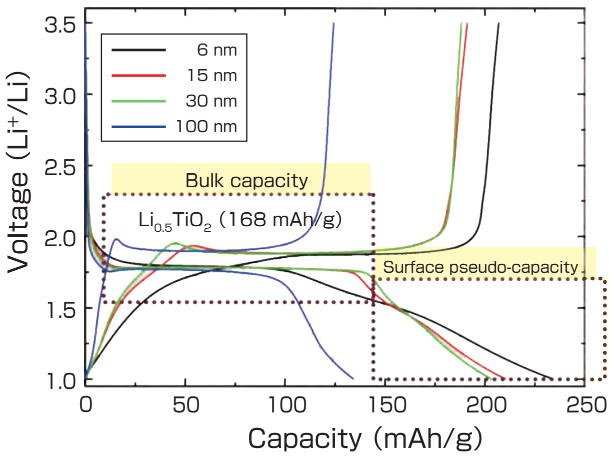

Fig. 6 Charge-discharge curve of nanocrystalline titania.

charge-discharge curve, as shown in Fig. 6. In nanocrystal titania, charge-discharge curve differed greatly from that of bulk material. The intercalation capacity in ordinary titania solid was normally 168 mAh/g for equilibrium composition $Li_{0.5}TiO_2$, whereas this increased to about 230 mAh/g in the research result. This capacity increased with smaller crystal size. It was thought that increase in capacity occurred due to lithium stored on the nanocrystal surface as pseudo-capacity on the surface in addition to intercalation capacity.

In fact, as shown by the charge-discharge curve, lithium was stored at certain site potential up to $Li_{0.5}TiO_2$, which is theoretical capacity of lithium composition, so discharge curve showed constant voltage of about 1.75 V. When this lithium composition was surpassed, pseudo-capacity discharge curve appeared with gradual decline in voltage along with increased capacity. Large pseudo-capacity that appeared in region below 1.75 V was apparently oxidative-reductive capacity of titania, and might represent the capacity of lithium that was electrochemically adsorbed to the surface of active material. These did not have constant site potential, and the voltage was thought to decrease, as the surface turned metallic as lithium concentration on titania surface increased.

On the other hand, the most important issue when designing high-power active material was question of most appropriate nanosize. From the result of size dependency of Fig. 6, when crystal size of titania was 100 nm and 30 nm, the electrode property increased, and the effect of nanosizing became apparent for active material of 30 nm size. Moreover, nanocrystal property became dominant in 6 nm size active material, different from the bulk material. From these results, there was great possibility in using active material with size less than several 10 nm when utilizing innovative nanocrystal property.

For charge-discharge mechanisms using pseudo-capacity on titania surface, high-speed charge transfer became possible since they did not involve slow diffusion process inside the solid. As shown in Fig. 7, intercalation inside the nanocrystal showed good electrochemical property for battery material since plateau voltage occurred in two-phase coexistence. However, this storage mechanism was not suitable for high-speed charge-discharge since it accompanied slow charge transfer process due to diffusion within solid. On the other hand, surface pseudo-capacity mechanism by adsorption and desorption of ion on the surface may enable charge transfer and lithium storage in second order since they did not accompany diffusion process. In fact, the change of discharge capacity when charge-discharge current density of titania crystal with anatase structure of 6 nm and 30 nm were changed is shown in the figure. In both cases, intercalation capacity tended to decrease as the current density was raised, and while capacity decreases severely in titania of 30 nm crystal size, titania with 6 nm crystal size showed little decrease under condition of large current density of 10 A/g, and we found that high-speed intercalation was taking place within the crystal.

In charge-discharge curve, capacity decreased severely in the flat part of voltage in 30 nm crystal, and it was found that the intercalation of lithium ion inside the crystal was inhibited under condition of large current density. On the other hand, when pseudo-capacity mechanism of nanocrystal surface was used, the pseudo-capacity at surface did not decrease much even when the current density was raised. This showed that charge transfer at surface reaction occurred at extremely high speed. In 6 nm nanocrystal, the pseudo-capacity content of the surface did not decrease even at high-speed charge-discharge to 40 A/g, and this implied that lithium ion possessed reversible high-speed storage property. That is, in nanocrystal active material, pseudo-capacity appeared due to large specific surface area, and lithium storage at over stoichiometric composition became possible. Moreover, new electrode property where high-speed lithium storage mechanism appeared at surface without intercalation into the solid was discovered. Using this energy storage property unique to nanocrystal, it would be possible to realize innovative high-capacity high-power lithium battery electrode material.

In industry-academia-government vertical collaboration development, it was necessary to synthesize nanocrystals using active materials, for which the battery manufacturer was considering product realization, and to investigate their high output property. If nanocrystals were synthesized using active materials used in the products and their high-capacity, high-power, and high-cycle properties were demonstrated, it should lead directly to practical use. To utilize the concept of nanocrystal active material in product development of batteries for power tools by Hitachi Maxell, one of the participant of this project, we developed the fabrication process of nanoporous structure and nanocrystal synthesis of lithium titanate $Li_4Ti_5O_{12}$ from which high output property could be expected among titanium oxide materials[4][5]. $Li_4Ti_5O_{12}$ active material is electrode active material with negligible level of crystal structure change in insertion and extraction of lithium ions, and therefore has drawn attention as electrode material with excellent charge-discharge cycle property. Therefore, AIST added polymer as dispersing agent for introducing mesoporous structure when synthesizing the active material, fabricated nanoporous structure electrode composed of framework of sequential mesopore and nanocrystal active material, and conducted evaluation of high output property.

To introduce mesopore with high ion diffusability to nanocrystal electrode body, template polymer P123 (Pluronic; EO20PO70EO20) was added during sol-gel synthesis of $Li_4Ti_5O_{12}$ electrode to promote dispersal of nanocrystal $Li_4Ti_5O_{12}$. After adding polymer to primer, and firing in air for 6 h at 400 ºC and then for 2 h at 750 ºC, we fabricated nanoporous structure electrode where $Li_4Ti_5O_{12}$ particles with size about 60 nm were linked in highly dispersed manner. When the electrode properties were evaluated, as can be projected from the concept of low-resistance, high-ion diffusion electrode, we obtained sufficient electrode capacity at high charge-discharge current density for $Li_4Ti_5O_{12}$ electrode with nanoporous structure, and the cycle property was good.

According to AIST research, it was found that the output

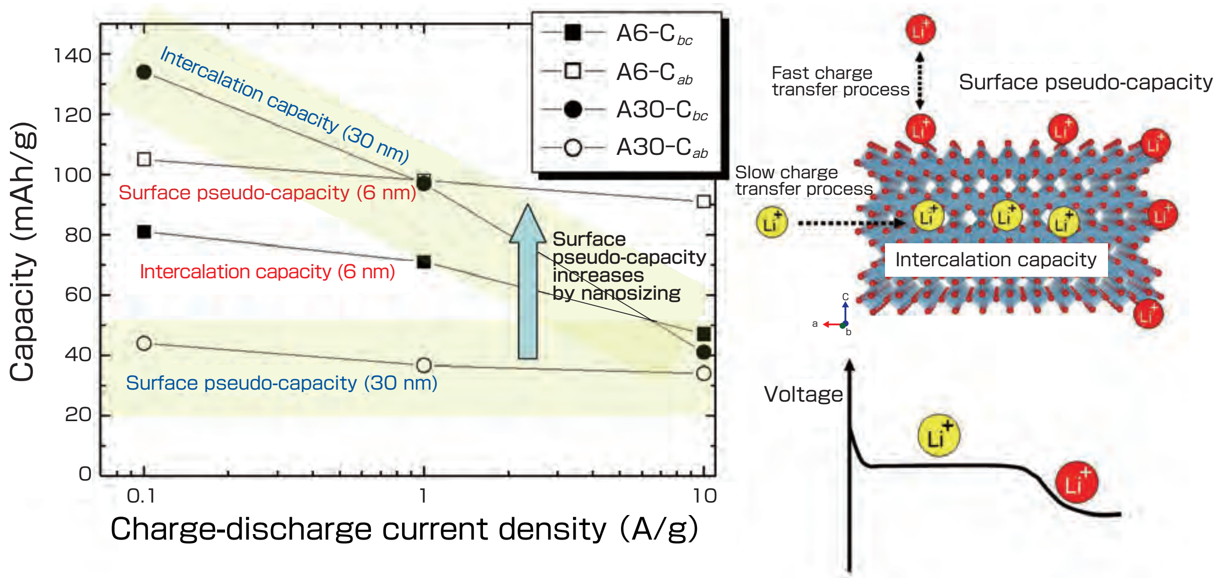

Fig. 7 Pseudo-capacity mechanism of nanocrystalline titania surface and its high-rate charge-discharge property.

property improved by adding nanoporous property to active material size of several 10 nm in $Li_4Ti_5O_{12}$ with high possibility for high-power negative electrode. In titania (TiO_2), which is similar titanium oxide, we studied the electrode property to 6 nm size, and clarified the energy storage property characteristic of nanosize material such as high-speed charge-discharge property and pseudo-capacity from the perspective of basic chemistry. On the other hand, existing battery products used bulk (μm level) size electrode, and there was no product realization using such small active material. Therefore, there was no systematic study on active material size for optimizing the battery power property in either industry or academia. Therefore, it is necessary to continue industry-academia-government collaboration research to investigate which nanosize shall be set as goal for nanotech electrode in product realization phase, or to constantly keep in mind what is the optimal active material size for high output property.

This was an important technological item for linear connection of R&D by university, AIST, and battery manufacturer. As shown in Fig. 8, there was unexplored area or “missing region” of size between the size regions explored for active materials in basic research by university and AIST and the size regions sought for the products by battery manufacturer. It was imagined that the optimal value for output property lies in this missing region. In vertical collaboration, it was important to study this size region from both basic and practical sides to quickly clarify optimal size, and to investigate the optimal solution in battery cell. In fact, region of active materials of several 10 nm to several 100 nm was not systematically surveyed until now, and R&D in the missing region was an important milestone in this vertical collaboration. In fact, as explained in the following example of Hitachi Maxell, battery prototypes were created using active materials of different sizes from 55 nm ~ 200 nm using

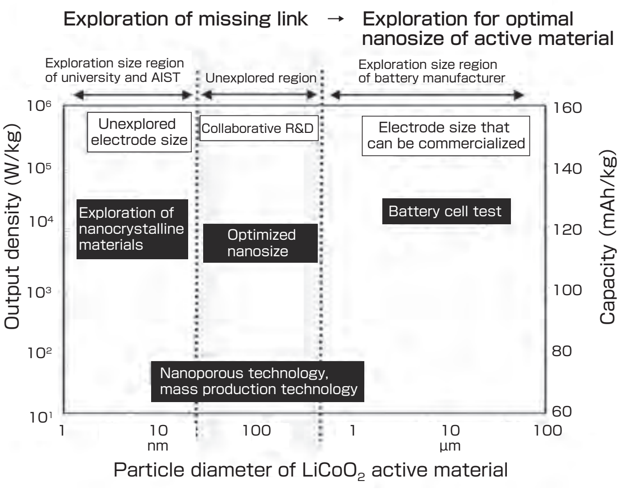

Fig. 8 Exploring active material size in the missing region to optimize the electrode property.

spinel structure manganese $LiMn_2O_4$ for practical electrode, and the output characteristic was evaluated. Active material size was shifted from bulk range to nanosize range in steps, and actual exploration was done on which size would be optimal for capacity and output within the missing region for intercalation electrode. This R&D project was probably the first systematic investigation of size dependency of electrode property for optimization of active material size for product realization, rather than just synthesis and property clarification of nanosize active material.

Next, in vertical collaboration, investigation of quick innovation potential of university and AIST was necessary. To investigate the potential for application to product using the excellent electrode properties of nanocrystal material that were learned from basic research, prototype creation and performance assessment of battery cells were conducted. Prototype fabrication was done by Hitachi Maxell that fabricated and evaluated standard spec laminated cell.

First, I shall describe the fabrication of the prototype battery. The electrode body used in the experiment was fabricated by mixing electrode active material, conductivity aid, and PVDF dispersion fluid binder, that were each weighed to target composition, in planetary ball mill, and paint-form electrode sol was fabricated after adjusting the viscosity. This paint-form electrode sol was applied using an applicator on of 15 μm thick aluminum foil so the dried weight would be 5.0~7.0 mg/cm^2 for both positive and negative electrodes, and the electrode body was finished by pressing after drying. The structure and photograph of laminated battery cell are shown in Fig. 9.

Considering the result of high output property of nanocrystal electrode at AIST, prototype cell with nanosize electrodes for both negative and positive electrode was fabricated. Negative electrode was made of 100 nm $Li_4Ti_5O_{12}$, while positive electrode was made of $LiMn_2O_4$ nanocrystal active material of different sizes from 55 nm to 200 nm, for purpose of finding optimal active material size in the missing region.

Although there were some variations in $LiMn_2O_4$ for active material size 150 nm or less, no significant decrease of discharge capacity occurred to 2 A/g rate. From active material

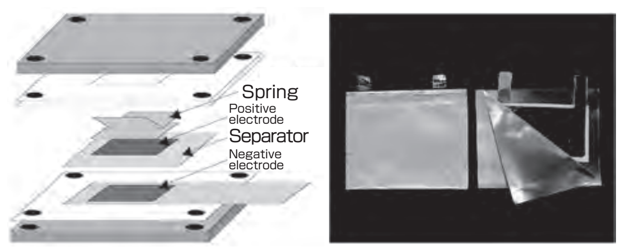

Fig. 9 Structure and photograph of laminated battery cell prototype.

size dependency of capacity retention, the property increased under high-speed charge-discharge condition of 5 A/g or over when the particle size decreased, and active material with 55 nm size showed best output property (Figure 10). According to AIST's calculation based on diffusion theory, for particle diameter of 100 nm or less, lithium ion diffusion within the particle would be sufficient for 100 charge-discharge per hour. Looking at the actual prototype cell data, when electrode using active material size of 150 nm or less was used, charge-discharge was possible even at high current density, and it was found that output property improved as active material size decreased. As result of optimizing the active material size in the missing region, best output property was obtain in battery cell using negative electrode (100 nm) of nanocrystal active material $Li_4Ti_5O_{12}$ with spinel structure and positive electrode (55 nm) of $LiMn_2O_4$. Its high output property was demonstrated at battery cell level using the innovative energy storage mechanism of nanocrystal electrodes. Finding the optimal size for nanocrystal active material to be used in high output battery was the most important issue since the commencement of the project. While the development of high-power battery using nanosize active material is becoming fierce around the world, to find out were the optimal solution lies in the 1 nm ~ 100 nm size range will solidify the foundation of innovation in storage technology.

In this R&D, AIST clarified by experiment that nanosize active material was effective for realizing high-capacity and high-power properties through its characteristic lithium storage mechanism. Moreover, by engaging in systematic exploration of missing region through vertical collaborative development for quick practical application, and by evaluating the output and cycle properties of the prototype battery, it was clarified by experiment that optimal solution was active material of size around 50 nm. Currently, while R&D of high-power lithium secondary battery using nanosize active material is accelerating around the world, this was the first R&D that specifically pursued which active material size was optimal for battery products. In the vertical collaboration project lead by the Author, optimal active

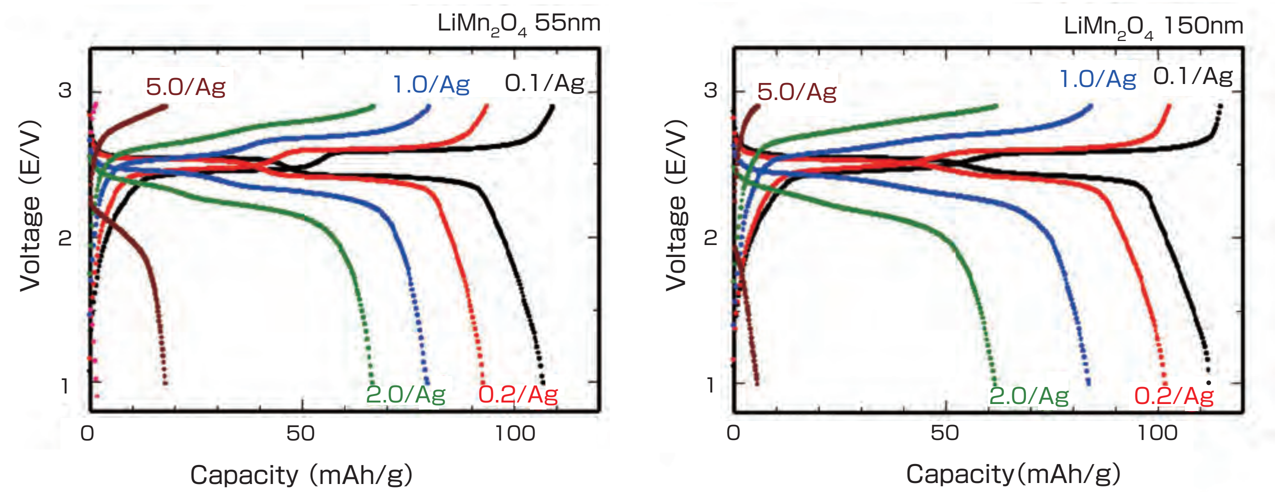

Fig. 10 Output property of lithium battery using nanocrystal electrode.

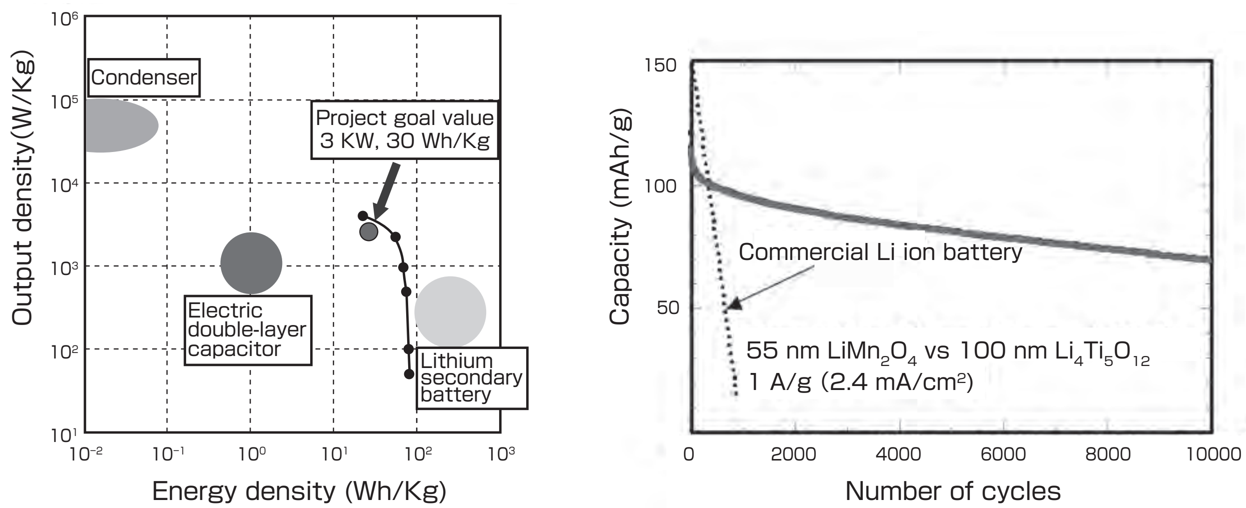

Fig. 11 Ragone plot of battery performance of prototype and charge-discharge cycle property (higher performances compared to existing batteries were observed for both items).

material size was found for product specification as result of systematic exploration of missing range of active material size in order to create innovative battery product quickly using excellent lithium storage property of nanosize active material that was found by university and AIST. Moreover, we fabricated the prototype battery cell was fabricated, and high output and cycle properties were confirmed to be applicable as product.

Figure 11 is a Ragone plot of the battery performance of prototype fabricated for the project. It surpassed the performance of existing lithium secondary battery products, and achieved high capacity and output properties that were set as project goal values. In addition, charge-discharge cycle property, which is the most important specification in product realization, was investigated to 10,000 times under high output condition, and capacity retention of 60 % was obtained. Hence, its superiority compared to existing battery products was shown, and it was found that it had reliability that could withstand product realization. Currently, product development of high-power battery utilizing the innovative energy property of these nanocrystal active materials is in progress. AIST generated the concept of nanocrystal electrode as innovative energy storage material, realized high output property at basic research phase, academically clarified the innovative energy storage property that arose from surface effect and size effect, and then demonstrated high-power lithium battery utilizing the excellent electrode properties of nanocrystal active material through collaboration with battery manufacturer.

4 "Shortening distance" to innovation through interdisciplinary fusion and industry-academia-government vertical collaboration

As strategy for "shortening distance" to innovation as described in the paper, Full Research was accelerated by interdisciplinary fusion and vertical collaboration of industry-academia-government, as shown in Fig. 12. I shall emphasize the point that characteristic of AIST is that innovative new technology is often generated through interdisciplinary fusion. Since it has high number and density of researchers, and has wide research spectrum that covers almost the entire range of industrial technology from standard, geology, bio, to information, fusion of diverse disciplines and integration of elemental technologies can be done readily. This means that it is a research organization with high potential for innovation appropriate for creating new technologies and concepts never seen before. Utilizing this organization structure, it is capable of generating innovative sprout technology in the interface of the disciplines. That is, bud of new technology can be created in diverse interface regions such as bio and energy, standard and nanotech, or electronics and environment, and high innovation potential that will serve as platform of innovative technology can be created.

Next point to be considered is the methodology of how to investigate the efficacy of such innovation potential in short time, or synthesiology of innovation. Taking as example the R&D for high power lithium battery, which the Author lead in the NEDO project, I shall discuss the R&D process that can investigate the efficacy of innovation potential in short time, for example in few years, through the vertical collaboration scheme of industry-academia-government. As shown in Fig. 12, vertical collaboration is organized by gathering the university (located upstream) that engages in basic research, AIST that engages in Full Research, and battery manufacturer and automobile manufacturer who is the end user (downstream) into one project that is vertically

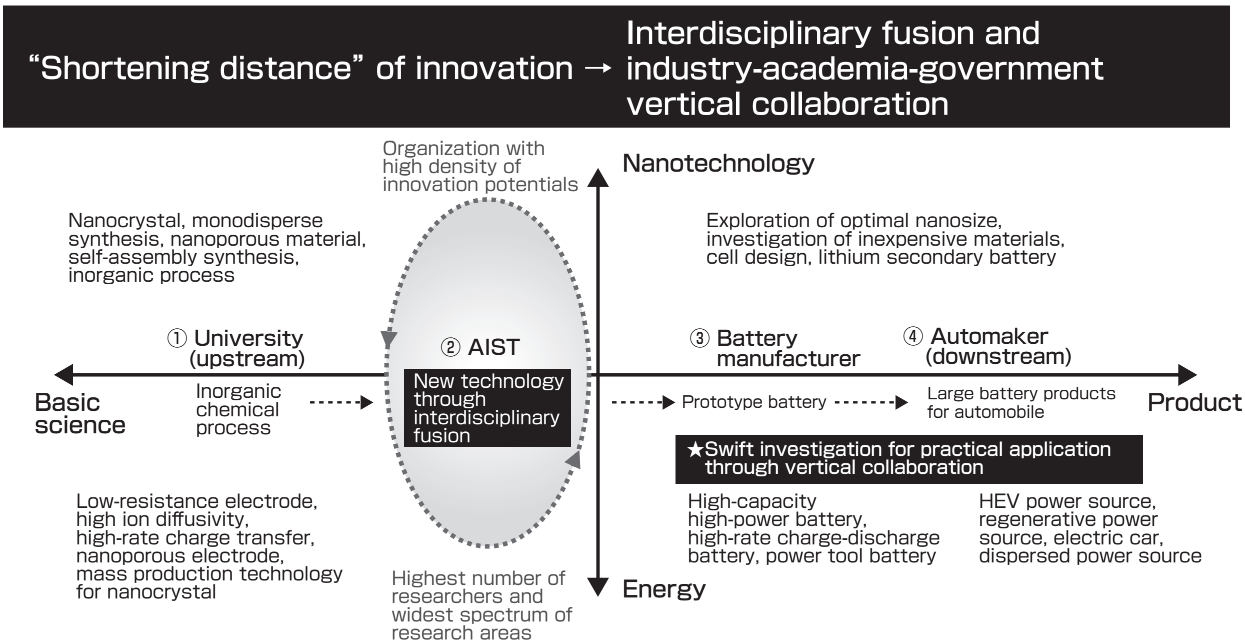

Fig. 12 *Synthesiological* method for innovation by industry-academia-government vertical collaboration.

linked, to push the innovation potential quickly to realization. In this collaboration scheme, the new technology of university and AIST is transferred quickly to the automaker, which enables quick investigation of efficacy and reliability. Technological request of the automaker can be back cast (fed back) to AIST and university through the battery maker, and this may provide direction for the basic research. By sharing this bi-directionality among the participating organizations, close information exchange and basic research that matches the product specification will be possible, and may lead to effective R&D process that enables practical result in short period of few years.

Next, what is the role of AIST in such new R&D process? In short, AIST can function as central innovation hub in such vertical collaboration. It is capable of creating innovation potential effectively and efficiently through interdisciplinary fusion because of the high number and density of researchers and wide spectrum of research fields. To find subjects that can be applied to product amongst the new technologies generated by interdisciplinary fusion, the industry-academia-government vertical collaboration project is effective as explained in this paper. As shown in Fig. 12, the process of quick technological transfer of innovative technology created by interdisciplinary fusion to product that has been set as clear goal using the industry-academia-government vertical collaboration scheme is an effective convergent R&D process that enables “shortening distance” to innovation. AIST functions as central innovation hub in this process.

In this paper, it was demonstrated that convergent R&D process was successful in innovation of high-capacity high-power lithium secondary battery, which is key technology for HEV power source. In synthesiology of innovation where collaboration is assumed, there are two methods: (1) vertical collaboration development as convergence process where clear product goal is set and various elemental technologies are integrated, and (2) horizontal collaborative development as co-creation process where diverse new technology is created without setting clear product goal, as shown in Fig. 13. In the NEDO project for which Author was the R&D leader, there was clear goal of developing a specific product i.e. high-power high-capacity lithium secondary battery for HEV, in shortest time possible. Therefore, the former convergence process was employed, R&D was conducted under industry-academia-government vertical collaboration scheme that involved university and company, and quick realization of innovation potential was attempted through fusion of nanotech and energy fields. The convergence mechanism that effectively integrates elemental technology is most appropriate to achieve clear product goal in “shortest distance,” and it was actually possible to develop innovative high-power battery in short period.

In conventional collaborative process, the idea of basic research from university is transferred to AIST, technological development is conducted right up to turn-over to industry such as measurement of performance and mass production process, and then technology is transferred from battery manufacturer to automakers. However, in such successive collaborative development, when the direction of R&D shifts according to the interest of the researchers at each

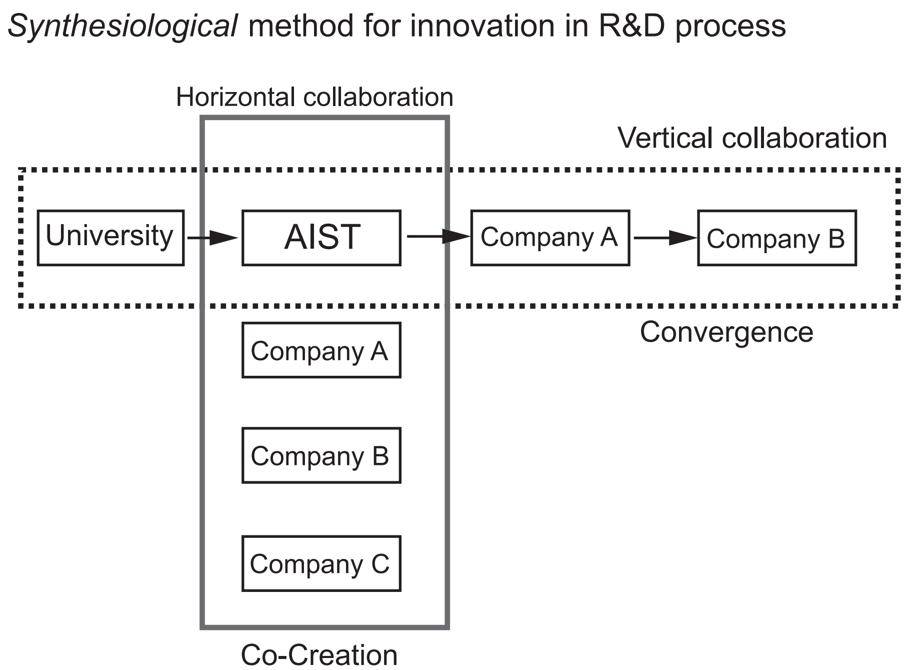

Fig. 13 Two-dimensional *synthesiological* method for innovation.

organization, the end user downstream may receive some technology quite different from one they were expecting. In this industry-academia-government vertical collaboration, the direction of basic research was set by participation of all organizations at commencement of the project and the technological development demanded by the automaker was fed back to university and AIST, to speed up downstream technological transfer that usually takes time and to ensure accurate technological transfer needed for the product. Distance between upstream and downstream was "shortened" by maintaining straight passage of technology flow. We succeeded in creating extremely hopeful innovative material, nanocrystal electrode, in three-year development period, and were fortunate to be able to develop innovative technology and built bridge between nanotechnology and energy technology that are generally considered difficult to join. Although the project achieved sufficient performance as power source for HEV, the product development of small cell is still in progress, using nanocrystal electrode for power tool that can be commercialized in few years.

Figure 13 shows the synthesiological method of innovation that are categorized as R&D processes, and convergence mechanism with vertical collaboration as explained in this paper is effective for product for which final goal is clear. For realization in short time period, it is most efficient to converge technology into the final product by integrating various elemental technology, and under vertical collaborative system, the technology transfer can be accomplished in shortest time period. On the other hand, if the goal is to create innovative sprout technology that does not exist or to seek diverse and highly generalized technological standard, co-creation mechanism with horizontal collaboration is better. This can generate diverse innovation potential and contributes to wide-ranging industry in horizontal manner. Although this paper described the example of convergence mechanism, both mechanisms are important as *Synthesiological* method of innovation at AIST. It is necessary to continue refining the methodology.

5 Future issues

Figure 1 shows the overview of the batter industry, and for application to automobile power source, upsizing process, safety, and cost performance are required at high level in addition to capacity and cycle properties. Since the development period of this research was only three years, we did not reach realization of automobile power source. The vertical collaboration scheme is mechanism that enables swift and sufficient technological transfer to all collaborating organizations. On the other hand, there is still very high hurdle in directing research through feedback from automaker to university and AIST and straight technological transfer from basic research to automaker. For upsizing battery, there will be no technological leap forward from small size level of cell phones and laptops to large battery for cars, but the technology will develop through several intermediate phases. In that sense, to create innovations, it is necessary to conduct industry-academia-government vertical collaboration that takes in consideration realistic conditions such as limited time and budget, as well as market strategy and technological potential of participating companies.

6 Summary

As diversity and speed are demanded in innovations and various collaborative R&D are sought, this paper discussed the convergent *Synthesiological* method of innovation by interdisciplinary fusion and industry-academia-government vertical collaboration as effective R&D process, using the example of high-power battery development. Based on the chemical synthesis process created at the university, AIST succeeded in developing nanocrystal electrode, which is high-performance active material that realizes high capacity and high output properties. As result of collaborative development with battery manufacturer to apply the active material concept to products, it succeeded in fabricating prototype of high-performance lithium battery with 30 Wh/kg and 3 kW/kg, which are performances demanded for regeneration power source for HEV. Moreover, superior cycle property needed for product realization was obtained compared to current product. Currently, R&D of product that will be commercialized in few years as power tool battery is in progress to utilize the result of this vertical collaboration development at participating battery manufacturer. Nanocrystal electrode is innovation of storage technology that was born from the fusion of nanotechnology and energy technology, and industry-academia-government vertical collaboration was a scheme appropriate for achieving strategic goal in short time through swift investigation of efficacy of innovation potential.

As discussed in this paper, creation of innovation potential that cannot be done by university or industry alone can be done easily at AIST, which is a consolidated research center that is capable of becoming core research institute in collaborative research. The reasons are because there are many researchers and wide research spectrum exists within one organization, and AIST can create diverse innovation potential highly efficiently through fusion of various disciplines. It was also demonstrated that R&D in vertical collaboration with participation by end user company was effective to swiftly check the possibility of application to target product. The new *Synthesiological* method of innovation (interdisciplinary fusion plus vertical collaboration) is an extremely effective scenario in speeding up R&D. In the R&D for high-power lithium secondary battery using nanotechnology, it was possible to develop the nanocrystal electrode, which is innovative material technology, to product realization phase in short time of three years. This scenario is also effective R&D process in other industries such as bio, information, nanotech, manufacturing, environment, and energy, particularly in short range project where the final product goal is clear.

7 Acknowledgements

I thank all people who cooperated in this industry-academia-government vertical collaboration development for high-power lithium secondary battery. Particularly, Zhou Haoshen, Group Leader of Energy Interface Technology Group, AIST contributed greatly for the property data when titanium oxide nanocrystal material was used as electrode material. Tetsuichi Kudo, Professor Emeritus of The University of Tokyo gave us essential idea for the concept of high-speed charge transfer that takes place within active material. I am grateful to the people of Hitachi Maxell, Ltd. for fabricating the prototype laminated battery and testing the battery properties. We received advice from Professor Moriguchi of Nagasaki University on the synthesis of porous electrode structure. I am also thankful to people of Nanotech Division, NEDO and Collaboration Promotion Department, AIST who gave us various advices in conducting this project.

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Discussion with Reviewers

1 The flow of vertical collaboration

Question and Comment (Koichi Mizuno)

Joint research in vertical collaboration style is method that has been addressed frequently. Can you indicate if there was any reverse transfer of R&D from the company to AIST, since collaborative research is not mere one-way flow from AIST to the company (battery manufacturer)? Of course, I see that the main topics of development were suggested by the company, but I think it will help explain vertical collaboration if you explain any bi-directional exchange of technological contents.

Answer (Itaru Honma)

AIST selected titania TiO_2, which was appropriate material for basic research since it was available in various sizes, to academically clarify the nanosize effect of electrode material, and systematically studied the electrode property of this oxide. In the basic research phase, we studied the appearance of surface capacity accompanying nanosizing, clarified that the nanocrystal active materials were suitable for high-speed charge-discharge, and explored optimal nanosize. Joint development was done by communicating to the companies in this industry-academia-government project the fact that excellent high capacity and high output properties of nanosize active material could be used in actual active material.

In fact, Hitachi Maxell was using $Li_4Ti_5O_{12}$, which is similar titanium oxide to titania on which AIST was working, for negative electrode material, but to downsize this material to nano level and optimize capacity and output properties, AIST and companies pointed out the necessity for clarifying which $Li_4Ti_5O_{12}$ size was optimal for use in commercial battery by exploring the missing range of active material size, as shown in Fig. 8.

Optimal nanosizing of practical electrode material listed in the commercialization plan was conducted through bi-directional information exchange and development plan of technological transfer of sizing effect of titania (fore-cast from AIST to Hitachi Maxell) and practical exploration of nanosize active material (back-cast from Hitachi Maxell to AIST). The 55 nm $LiMn_2O_4$ and 100 nm $Li_4Ti_5O_{12}$ used in prototype cell data were results for optimal nanosize found in this collaborative development, and as described in the paper, output property that far surpassed the conventional battery performance was obtained.

2 Surface pseudo-capacity

Question and Comment (Koichi Mizuno)

The "pseudo-capacity" on surface is the key point in increasing charge-discharge capacity rather than intercalation inside the electrode solid, and I think it is a good example of application of nanosizing of active material. Although it is described that in surface pseudo-capacity, “the surface becomes metallic as lithium concentration increases and voltage decreases,” it may be helpful if the author explain the reaction mechanism and specific image of the surface related to the pseudo-capacity.

Answer (Itaru Honma)

To provide simple explanation, as shown in Fig. 7, lithium ion causes intercalation (insertion of ion) inside the solid in bulk reaction, and at the same time, oxidation reduction of the compositional metal occurs to store electric energy. It arises from the essential (perhaps standard?) electrochemical reaction of the material. Therefore, no matter who conducts experiment anywhere in the world, as long as material with same stoichiometric composition is used, same amount of lithium ion is stored in equilibrium voltage. However, the storage mechanism at surface is different from the essential (bulk) property unique to the material, and it is non-unique property that manifests because a surface exists. Therefore, difference occurs in capacity property due to change in specific surface area or plane direction (crystal morphology) (that is, data will differ according to experiment group).

In Figure 7, it is described as surface reaction where electron enters the titania surface layer and causes single-electron reduction of Ti^{4+}/Ti^{3+} at same time as adsorption of lithium ion to the surface in the fast charge transfer process. That is, regardless of crystal structure, it is thought that pseudo-capacity originates from surface reaction. Moreover, the two-electron reduction of Ti^{4+}/Ti^{2+} that does not occur in bulk becomes possible in nanocrystal surface with special chemical bond status for lithium ion, and that produces large capacity. The lithium ion shown in red in Fig. 7 is presenting special surface reaction, and shows that the lithium is stored at higher concentration than bulk. If two-electron reduction takes place, Ti^{2+} will appear, so voltage decreases and the surface becomes more metallic as shown in bottom figure.

In addition, grid expansion occurs in nanocrystal and crystal structure (phase) different from bulk becomes stable, and the surface is not necessarily same as surface of bulk. These are extremely interesting research subject. In other words, there is possibility that new storage mechanism may appear, and we intend to pursue this basic research further.

3 Collaboration of AIST with battery manufacturer and automobile manufacturer

Question and Comment (Akira Ono)

I think this research demonstrates advantages of the vertical collaboration of industry-academia-government. Then, I ask the Author, who was the project leader, about the collaboration. In this project, what specific suggestions did AIST receive from the battery manufacturer, and how were they reflected in the project? Also, what specific suggestions did AIST or the battery manufacturer receive from the automobile manufacturer, and how were they reflected in the project? Please answer from the standpoint of project leader.

Answer (Itaru Honma)

It has been found that high output can be obtained using nanosize active material and basic research is accelerating in this area, but I was surprised that there was absolutely no research on which nanosize materials were optimal for $LiMn_2O_4$ and $Li_4Ti_5O_{12}$ that are practical electrode materials. As shown in Fig. 8, there is missing range in active material size, and the battery manufacturer suggested that we should seek physicochemical knowledge of nanosizing effect by exploring this unexplored range, and search for sizes that generate highest capacity and output properties, as most important items of development. It is important from perspective of basic research to systematically investigate the size effect of active materials in nano range, and the surface effect that becomes clear in the process and size effect of charge transfer process of ion and electron in that process become very important guides for material design in developing high-power electrode. AIST set direction of basic research to respond to the expectation of the battery manufacturer, and studied the nanosizing effect of metal oxide materials such as titania, as described in the paper. The automobile manufacturer indicated that low cost and productivity are important issues in actual commercialization, and we worked simultaneously on mass production process of nanosize active material based on this suggestion. Although not included in this paper, we developed new active material synthesis process where nanocrystal active material could be mass-produced in kilogram level using molten salt method.

4 Collaboration of AIST and university

Question (Akira Ono)

What specifically was the collaboration between Nagasaki University and AIST? Rather than transferring just the technological potential of Nagasaki University to AIST, can you talk specifically about the interaction and coordination between the two organizations from the standpoint of project leader?

Answer (Itaru Honma)

Nagasaki University (NU) made major contribution in developing the basic chemical process. AIST has been studying the nanosizing effect of active material, and NU selected the reaction process appropriate for synthesis of the material. Specifically, they conducted reaction kinetic investigation for the hydrothermal synthesis and molten salt synthesis methods used in this project, and we received basic but very important advices on starting material and solvent types. Moreover, they conducted basic research on low cost synthesis that was requested by the automobile manufacturer. Particularly NU investigated the practical process where synthesis could be accomplished as a one-step firing process for carbon high-capacity electrode material. The result is being utilized in battery development by the automobile manufacturer.

In the collaboration of AIST and NU, to maximize each other’s research potential in extremely short period of three years, the development of nanocrystal active material of metal oxides was done by AIST, while the development of nanoporous and high-power carbon materials that are essential as conductivity aid in actual battery electrode was done by NU. By doing so, we were able to design innovative high-capacity high-power electrode through combining the two results during the course of project.

From basic research on firefly bioluminescence to *Product Realization Research*

— Production of a multigene expression kit based on bioluminescent proteins —

Yoshihiro Ohmiya* **and Yoshihiro Nakajima**

[Translation from *Synthesiology*, Vol.1, No.4, p.259-266 (2008)]

In the postgenome era, biological network analysis has become essential for elucidating animal physiology. We have developed a revolutionary tricolor reporter *in vitro* assay system that can be utilized for detailed analysis of a biological network. Bioluminescence has potential for visualizing the dynamics of living systems. To accomplish our vision, we are realizing new technologies through *Type 1 Basic Research*.

Keywords **:** Product Realization Research, bioscience, biotool, optical technology, gene analysis

1 Introduction

Bioluminescence refers to light produced by bioluminescent organisms such as the firefly. Light-emitting organisms possess a light source and an enzyme that catalyzes the luminescence of the light source. In light-emitting organisms, the light source is luciferin (meaning something that is luminescent or emits light) and the enzyme that catalyzes the reaction is luciferase. The French scientist Raphaël Dubois coined the terms luciferin and luciferase in the 19th century, and many researchers have studied bioluminescence science thoroughly. The field of bioluminescence science is not dominated entirely by European and American scientists, and the Japanese have contributed greatly to this field. For example, before the World War, Sakyo Kanda, who is often considered the second Minakata Kumagusu, wrote *Hotaru (Firefly)*, a work widely recognized both in Japan and overseas. Dr. Yata Haneda, the first director of the Yokosuka City Museum, was a world-class bioluminescence scientist, and Dr. Osamu Shimomura of the Woods Hole Marine Biological Laboratory is the discoverer of green fluorescent protein (GFP), which revolutionized bioimaging technology[1][2].

Since the 1990s, biotools using bioluminescence have been commercialized by companies in Japan, Europe, and the United States. However, because bioluminescence science is a composite discipline comprising wide-ranging fields including biology, chemistry, physics, biochemistry, and engineering, it failed to attract knowledge, technology, people, and money, and little innovative technological development has occurred. This was because of insufficient fusion of disciplines and the lack of a clear path through the "valley of death." Companies that have commercialized biotools using bioluminescence have not produced sufficient results from basic research to differentiate their products, to generate new products, and to communicate a positive message to the market.

In contrast, the Human Genome Project that started in the 1990s advanced rapidly by attracting knowledge, technology, people, and money, and decoding of the human genome was completed in less than 10 years. However, this advancement fell into the dilemma of sitting around waiting for the next discipline. The scientists initially believed that decoding the genome would provide groundbreaking basic information and great innovations would follow. Until then, it was believed that the human genome contained information for more than 1,000,000 proteins, and there was vague hope that this information would indicate the clear difference between humans and other animals. In reality, there are 21,600 genes in the lancelet, a chordate closely related to vertebrates, and the human has about 20,000 genes; thus, there is 2 % or less difference in genetic information between humans and monkeys. This raises questions such as, "Why are there differences between organisms?" or "Are humans really advanced?" There are now more issues in this postgenome era. Now, kinetic analysis and imaging of biomolecular groups that comprise life are gaining attention as methods to answer these questions.

Ohmiya, one of the authors, became interested in bioluminescence in the 1990s and engaged in research on the mechanism that determines the color of luminescence in the firefly, as part of the Sakigake 21 Project of the Japan Science and Technology Agency. He continued research on the biology and biochemistry of luminescence mechanisms of luminescent beetles and the sea firefly in the Faculty of Education, Shizuoka University. There, he succeeded in cloning the

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genes for red and green luciferase of Brazilian railroad worms and found that the 216th amino acid residue in firefly luciferase is involved in determining the color difference of luminescence[3][4]. Railroad worm (or glow-worm) is a luminescent beetle that survives only in Brazil, and expresses a luciferase that produces the strongest red-colored light on earth. Although these research results were new and may help produce biotools for bioluminescence systems, there was no direction that linked the results together. *Type 2 Basic Research* is not feasible for closing the gap between research and education within the university. In such background, Ohmiya and Nakajima started research at the new National Institute of Advanced Industrial Science and Technology (AIST), where they were given the opportunity to expand the knowledge gained in *Type 1 Basic Research* to *Type 2 Basic Research*. Development of biotools using the luminescent color differences started to take shape as an outcome, and a clear goal was set to link bioluminescence to kinetic observations and imaging of biomolecular groups that was being sought in 21st century biosciences.

2 What would be developed?

In 21st century bioscience after the completion of the Human Genome Project, it is understood that there are limited methods for pursuing a single biomolecule and that innovation would not happen by developing only measurement devices that are extensions of conventional methods. From this background, the "Development of Analysis Technology of Dynamism of Intercellular Network" Project started in 2002, lead by the Ministry of Economy, Trade and Industry, and New Energy and the Industrial Technology Development Organization (NEDO). The objective of this project was to measure efficiently the temporal and special kinetic changes in the intracellular biomolecular network that comprises the foundation of building and functioning of organic tissues in live cells and to establish the technology to allow this functional analysis. The project's goal was to clarify the information network created by several biomolecules. We participated in this project and suggested the use of luminescent proteins for the development of intracellular labeling technology of multiple types of biomolecules.

Our research concept was simple. Whereas traditionally, the amount of light was the only focal point in using firefly luminescence, we looked at the multicolor characteristic of firefly luminescence and studied how to transmit multiple bits of information from the cell. In other words, conventional tools using bioluminescence were an extension of black-and-white television, and we decided to use the color television characteristic, or color difference, to monitor the movement of multiple types of biomolecules in the cell. Our work would connect the results obtained in basic research conducted at the university in the 1990s. Figure 1 is a revised version of one of the slides used in the proposal and describes the research concept and strategy, and the use of luciferase for detecting diverse bioluminescence. Our powerful method was a first-in-the-world result of *Type 1 Basic Research* showing that the luciferase of railroad worms produces red light from the head and green light from the body.

The goal was to develop a multiple drug screening system that would emphasize clearly its difference from conventional technology (Figure 2). The product was ultimately commercialized in April 2006 as "TripLuc" from Toyobo Co.,

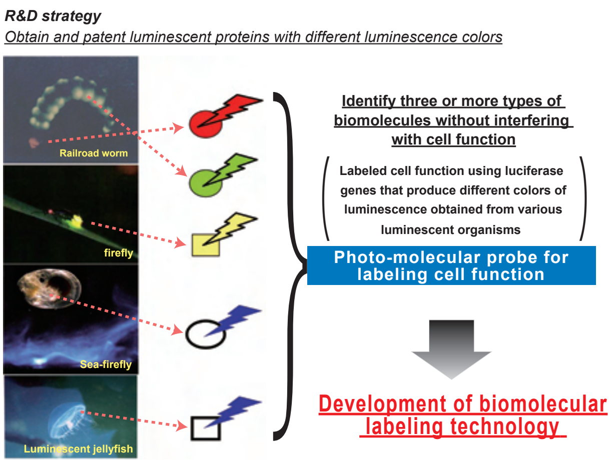

Fig.1 R&D strategy. Revised version of slide used at NEDO "Intracellular Dynamism Analysis" hearing.
The photographs on the left show, from top to bottom, railroad worm, Japanese firefly, sea-firefly, and luminescent jellyfish. The cell function labeling technology that we studied was accomplished using a photomolecular probe as the label that "identifies three or more types of biomolecules without interfering with cell function," based on luciferases (enzyme or catalyst for luminescence) with different colors obtained from various luminescent organisms.

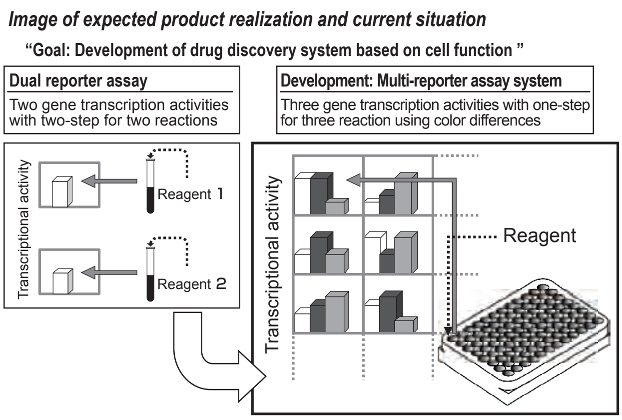

Fig. 2 Example of outcome of R&D. Revised version of slide used at NEDO "Intracellular Dynamism Analysis" hearing.
In conventional technology, two gene transcription activities were observed in two steps using two reagents, whereas in the newly developed multireporter assay system, three gene transcription activities could be observed in one step using one reagent. This allows the analysis of several samples at once (high-throughput analysis).

Ltd., and "MultiColor Luc" from Toyo B-Net Co., Ltd. We have sold the kits from these companies, as we had projected as an outcome of the proposal. The decision whether the company will sell the product depends on the demand for the products and the number of users and potential users that demand improvement in technology and expect the introduction of new technology. Japanese bioresearchers tend to take up technology they see in foreign journals rather than using their own judgment to introduce and adopt new technology. It is important for researchers to be alert to new developments and for those who produce new biotechnology to communicate the advantages of their products through as many channels as possible. Conducting *Type 2 Basic Research* and practical research that starts from a patent will not lead to true product realization unless it is followed up by continued aftercare, which produces solid results from *Type 1 Basic Research* based on the new technology created.

The labeling technology for studying multiple types of biomolecules involves monitoring the expression of several genes in the cell. The expression of several genes is regulated by quick or slow responses to external stimulus in the cell, which produces various proteins. For example, when an environmental hormone reaches the cell, the cell produces a female hormone in response. To detect the expression of a certain gene, we developed a reporter assay using the firefly bioluminescence enzyme, luciferase. In this reporter assay, the promoter region of the gene sequence that regulates the expression of the gene is inserted into the firefly luciferase gene and this is then transduced into the cell. If the gene expression is induced, luciferase is synthesized accordingly. Because luminescence occurs when luciferin is added to the expressed luciferase, the extent of genetic expression can be assessed by the amount of luminescence. This method was already employed in the biomedical field and has become a standard chemical assay in the environmental field (approved as an official method by the Ministry of Environment) and in screening methods for drug discovery. In 2002, the method was part of a 500 million-yen market in Japan and 20 billion-yen market worldwide.

Is it possible that these companies will realize their products in this field? The reporter assay that was supported widely until 2002 was the dual reporter sold by Company P. This product used a two-step measurement method comprising two genes and two substrates, and Company P, which held the patent in the United States, dominated the market. Companies trying to break this domination had to search for other potential reporter assay systems. Users were dissatisfied because the method could assess expression of two genes only, and there were problems relating to the cost and the two-substrate two-step process. Potential users wanted to a reporter assay whose application could be extended to more genes. We were able to set a clear goal to develop a new "three-genes, one-substrate one-step reporter assay" using the principle of the diversity of luminescence color difference.

3 Patent construction for product realization from *Type 2 Basic Research*

The light emitted by the firefly occurs through an enzymatic reaction in which the oxidation of firefly luciferin is catalyzed by firefly luciferase. There families of luminescent beetles are the Lampyridae (firefly), Elateridae, Drilidae, and Rhagophthalmidae (Iriomote firefly) families. These are unique in that their colors of luminescence differ slightly and because the luciferin–luciferase reaction color may be changed depending on the pH of the reaction environment. We decided to detect the multiple gene expression using luciferases that produced different colors but that were not influenced by a pH change. We have already isolated the luciferase genes that produce red and green light from the South American railroad worm, which produces varied luminescence (i.e., the head emits orange to crimson colors while the abdomen emits green to yellow-green colors). We have succeeded in expressing luciferase in E. coli and in producing orange luminescence using this genetic information[3][4].

However, when applying the multiple gene expression detection kit in 2002, we were unable to produce the stable expression of luciferase in mammalian cells. Although we

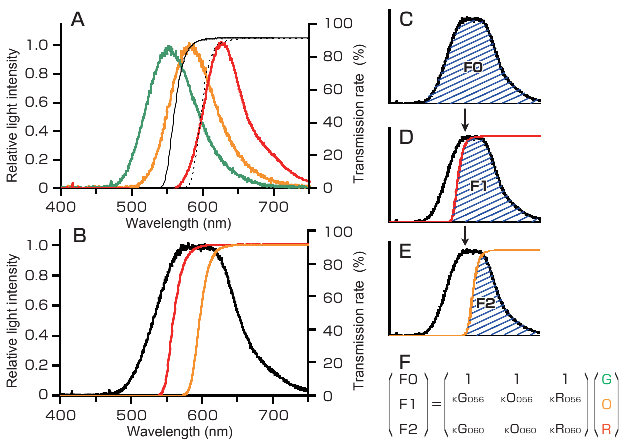

Fig.3 Outline of technology for dividing and measuring three luminescence colors using two filters.

A) Luminescent spectrum group of green, orange, and red luciferases.

B) Optical absorbance of two filters used and luminescence spectra when three colors are mixed.

C) F0 is the amount of light when all-optical output is measured without a filter.

D) Most green light is absorbed when passing through the first filter. F1 is the amount of light not measured.

E) Most green and orange light is absorbed when passing through the second filter. F2 is the amount of light not measured.

F) Equation to calculate the original amount of luminescence by calculating F0, F1, F2, and the transmission coefficient of the two optical filters, which had been predetermined.

had the idea for the new kit, we were unable to write the example for the patent. Nakajima *et al* considered it essential to improve the efficiency of transcription and translation processes when railroad worm luciferase is synthesized in the cell. They altered the arrangement of the genetic sequence and succeeded in altering the gene structure of the enzyme that could be used in mammalian cells in 2003[5]. Although the spectra were stable without being affected by pH, the next issue was how to divide and quantify the overlapping spectra. Obviously, the spectra that do not overlap could be divided by filters and quantified, but this would measure the extremes of green and red, which would mean that only two colors could be handled and most of the luminescence would be wasted. Through the joint effort of AIST, technologists at ATTO Corp., and Associate Professor Akiyama of the Institute of Solid State Physics, we developed a technology to divide and quantify three luminescence colors using two filters without wasting any of the light[6]. In the newly developed technology, luciferase is measured without a filter and with several optical filters to determine the transmission coefficient when various filters are used for each luciferase. The amount of luciferase is calculated from the transmission coefficients after the measurements using the various filters (Figure 3). This series of research was the result of *Type 2 Basic Research*. Although much may be gained through by a great leap of ideas, it is important to accurately grasp the current technology available and to use the "supreme knowledge" available at that point. Rather than producing supreme knowledge, to use it appropriately is the approach of *Type 2 Basic Research*.

The above description explained the success of commercialization in 2005, and although it seemed that the practical application went smoothly, it was not that easy. We wanted to patent the concept quickly, and we wrote the patent when we succeeded with the three-gene, two-substrate method using red and green railroad worm luciferase and existing sea pansy luciferase. Under the title "Genetic construction composed by integrating either of the two luminescent protein genes that emit different color luminescence in same luminescent substrate so there will be stable expression in mammalian cells," we applied for a patent in 2003[7] and published the concept[8]. At that point, we obtained a one-year grace period, which gave us time to study other luciferases. We were finally able to add to the patent of "the three-gene, one-substrate method" by combining the green luciferase from the Iriomote firefly, which was another subject at the time, and its orange mutant[6].

In the patent construction strategy, we received advice from the patent attorney to, first, take advantage of the one-year period after the patent application and, second, to ensure that examples for the core of the patent were written by as few people as possible and that patents were filed individually. The concept was emphasized in the patent, and although the patent describes a range of examples demonstrating such applications, it avoided details that may interfere with the companies' free research activities and product realizations. In particular, we did not include a description of the reagent of the multiple gene expression detection kit, and we left room so that companies can continue research to create reagents for the kit. The concept that formed core of the entire technology was known only to AIST, but the practical patent was constructed by placing priority on the joint patent application through joint research with companies or solo application by a company. As result, the aforementioned two companies worked on product realization and were able to sell the kits under their brands. Because these two companies were biotechnology companies, they were strong in software but weak in hardware. Therefore, we introduced Company A, a measurement instrument company, to ensure smooth product realization.

4 What can the multiple gene expression detection kit do?

We will use the MultiReporter Assay System Tripluc® from Toyobo Co., Ltd. as an example to describe our system. The name of the product, Tripluc, derives from "triple color" (luciferases for three colors and "trip" (to travel into different dimension past the conventional). It represents the general image and reflects the good taste of the company. I believe that the researcher should refrain from suggesting a name for the product because the company marketing department has the expertise to create meaningful product names.

The Toyobo kit is a package comprising green luciferase from Iriomote firefly (λ_{max} 550 nm), orange luciferase that is a site-specific mutant of green luciferase (λ_{max} 580 nm), red luciferase from railroad worm (λ_{max} 630 nm), and a promoter sequence that acts as a control for the three enzymes. Ultimately, the company will developed and marketed the reagent. The kit divides the tricolor luminescence spectra and quantifies these using optical filters, allowing the simultaneous measurement of two or three transcription activities. Because these luciferases all use D-luciferin as the luminescent substrate, the detection reaction can be done in one step, and all measurement is made by a luminometer equipped with optical filters.

Gene expression is regulated by bonding of transcription factors to promoters and cis elements that are present near the transcription start point of the gene. The reporter assay using luciferase can assess gene expression by joining the sequence that regulates gene expression, such as a promoter, to the luciferase gene, introducing this to the test cell, and then measuring the activity of the expressed luciferase. Using this reporter assay, it is possible to analyze the function of the transcription regulation region of a target gene and the mechanism regulating its transcription factor. Conversely, the luciferase gene can be joined to the transcription regulation region of a gene whose expression changes in response to a certain phenomenon to analyze the signal transmission of that phenomenon or action as well as the mechanism of action of the receptors and ligands.

The multiple gene expression detection kit that we developed can simultaneously analyze the expression of several genes. The kit comprises vectors of tricolor luciferase genes to which a transcription regulation region can be inserted. The user can extract the transcription regulation region of the gene to be measured using some method and insert this in the vector of the kit. The inserted genes are chemically or electrically transduced into the cell or body of an animal or plant. For example, once the gene is transduced into the cell and the chemical substance of interest is added, the expression of the target gene is regulated by the stimulus, which changes the amount of luciferase synthesized. The cells are crushed, the luciferin solution optimized for the measurement condition is added, the luminescence of each color is measured, and the change in gene expression is calculated. Our patent covers the concept of analyzing three gene expression patterns using three colors and the tricolor luciferases. The optimized luciferin solution is covered by corporate patent.

Figure 4 shows the result of a model experiment that provided the measurements[6]. The roles of the bonding site of transcription activation factor (RORE sequence) within the clock gene *Bmal1* promoter sequence that exists in mammalian cells and the peripheral sequences were investigated in the model experiment. In the *Bmal1* promoter sequence, there is one RORE sequence and two similar sequences where the gene transcription activation factor RORα4 could bond, and we compared the roles of the *Bmal1* promoter sequence and an independent RORE sequence (Figure 4A). We created gene vectors with the RORE sequence and SV40 sequence to support the role of the sequence for red luciferase, the promoter sequence including the RORE sequence for orange luciferase, and the SV40 sequence, which acts as a control for green luciferase. These were genetically transduced into the cell (Figure 4B top). Adding the transcription activation factor RORα4 to the cell activated the *Bmal1* promoter and promoted gene expression in proportion to the amount of RORα4, but the luminescence of red luciferase, which represents the transcription activity of the RORE sequence did not increase and gene expression was not promoted sufficiently (Figure 4B). We had expected the sequence similar to the RORE sequence to play a major role in regulating the overall transcription. As seen in this example, the multiple gene expression analysis system enabled the simultaneous observation of the expression of three genes, which used to be a difficult process. We also succeeded for the first time in analyzing simultaneously the time course of expression of multiple genes within living cell[9]. The application of this system is not limited to biological clock analysis, which we investigated, and we expect this system to be applied in the fields of cell biology, pharmacology, and molecular physiology.

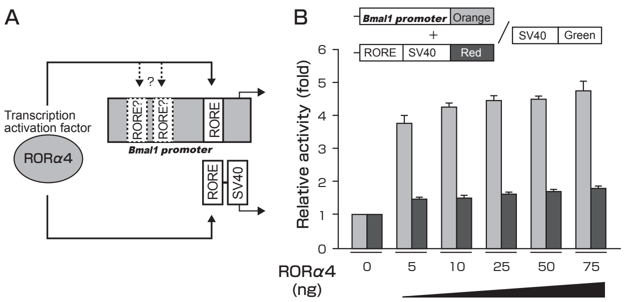

Fig. 4 Example of analysis of gene expression using multiple gene expression kit.

A) The clock gene *Bmal1* promoter sequence in mammalian cells contains one transcription activation factor-bonding site (RORE sequence) where the gene transcription factor RORα4 bonds and changes the amount of expression, and two similar sequences. The gene transcription activity of the RORE sequence can be assessed by extracting the RORE sequence individually and inserting it into the control promoter SV40 sequence.

B) Gene vectors are created with the SV40 sequence, which can support the role of the RORE sequence and the role of that sequence in red luciferase, with the entire promoter sequence including the RORE sequence in orange luciferase, and with the SV40 sequence that will serve as a control in green luciferase. These vectors are then transduced into the cell. Adding the transcription activation factor RORα4 to the cell promotes the expression of *Bmal1* in proportion to the amount of RORα4, whereas adding the RORE sequence only does not promote gene expression.

The multiple gene expression detection kit resulting from our study can be used to compare the responses of three or more genes. Highly reliable biological information can be obtained when assessing the toxicity of chemical substance as an alternative method to animal experimentation and for assessing efficacy in drug screening. We are currently working on a multicolor reporter system for toxicity assessment and trying to expand the use of this technology under NEDO's "Development of Hazard Assessment Method Using Cultured Cell" program. In the future, compatibility of data obtained from the simultaneous measurement of several samples will become important in toxicity assessment, and we are developing a new optical measurement device with high sensitivity and standardized optical metrology. Incorporation of new optical metrology technology will be very important for expanding the use of the multiple gene expression detection kit.

5 There is no end to the scenario of product realization

The basic concept of a multiple gene expression detection kit or "reporter assay for three-genes one-substrate using luciferase with different luminescent colors" has been put into practical use and realized as a product, and progress was made according to the scenario as a strategy. We are halfway through the scenario, and we have not reached the endpoint. The three issues to address to complete the scenario and our solutions are as follows. 1) Is this concept correct? We shall solve this through *Type 2 Basic Research*. 2) Can the companies realize the products? This will be solved through dialogue between companies that can produce the products. 3) Will the concept be accepted by society? The answer to this is perseverance. It is vital for both researchers and companies to transmit information actively. Further ideas about addressing these issues follow.

1) Is this concept correct? Even if we think it is correct in the beginning, ideas can fail. Many researchers, including us, have experienced failure, although most are never published, so we never know. The importance of *Type 2 Basic Research* is to go beyond literature searches to gather information from other researchers through discussions such as at seminars. This involves making full use of personal connections as researchers and will provide a method to apply the concept in practical use by knowing about the best technology currently available. The important point is not to generate supreme knowledge through ideation, but to use supreme knowledge to expand our understanding to solve particular problems. It is also important to span different disciplines. Because our kit produces light or luminescence spectra using tricolor luciferase, we needed to find a method of quantifying individual colored light. We did not have the knowledge to solve this problem, but it was solved easily by a physicist. Collaborative research across disciplines is a key to promoting *Type 2 Basic Research*.

2) Can the companies realize the products? Product realization can be accomplished by companies with sufficient technological ability if the companies find the concepts and results attractive, feel the patent is worth secure, and perceive that there are potential users in the market. It is important to approach the researchers of a company and to convince the intellectual property division, legal division, sales division, and, of course, management. *Type 2 Basic Research* results from the clear communication of the difference, novelty, superiority, and justification of the new method compared with the conventional method (e.g., technology, cost, and users). However, researchers should not obstruct product realization by other companies but should file patent applications independently and make logical decisions and good judgment according to the contract.

3) Will the concept be accepted by society? The researchers must focus on two aspects. First, they must work with the company to transmit information through, for example, lectures and reviews for seminars organized by the company. Second, the researchers must return to *Type 1 Basic Research* and create new knowledge that maximizes the technology they created. The researchers should help society understand and recognize the value of technology, and work should continue; that is, there is no end to the scenario.

Figure 5 summarizes these steps: *Type 1 Basic Research* develops into *Type 2 Basic Research*, the product is realized through collaboration with companies, and the researchers return to return to *Type 1 Basic Research*. In each step, two-way collaborations among researchers and companies are important.

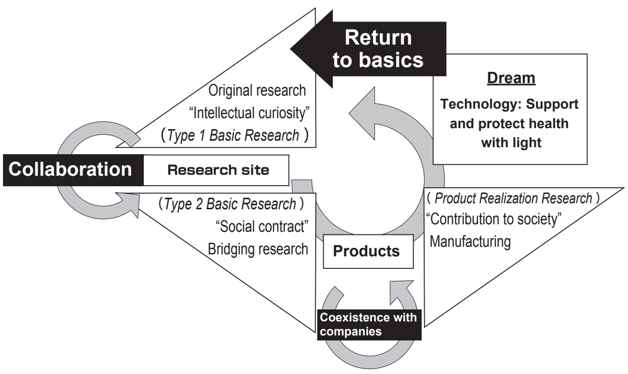

Fig. 5 Continuation of *Type 1 Basic Research*, *Type 2 Basic Research,* and *Product Realization Research* as perceived by the authors.
When *Type 1 Basic Research* develops into *Type 2 Basic Research*, the return from *Type 2 Basic Research* to *Type 1 Basic Research* is important. When *Type 2 Basic Research* develops into *Product Realization Research* with corporate collaboration, it is sometimes important to return to *Type 2 Basic Research* to shape the research results. Even after the product is realized, the research does not end, but returns to *Type 1 Basic Research*, which may take a new direction.

6 The struggle to realize the dream

Our purpose is to use light to extract biological information. In reality, the information we obtain is a shadow created by light, but it is possible to obtain useful information if that information can be extracted smoothly using light. From such perspective, we are dreaming of developing technology "to support and to protect health with light." Practical use of the multiple gene expression detection kit is the first step toward, and it is now possible to obtain highly reliable biological information through a diversity of bioluminescence, for example, through a high-performance simple hazard assessment method. We expect that increasing the reliability of the biological information obtained at the cellular level will provide alternatives to animal experimentation. It may also be possible to develop technology "to protect health with light." Looking at several aspects of biological information simultaneously provides a view of the cellular dynamism through light, which may provide new information about life "to support health with light." This approach uses *Type 2 Basic Research* to produce new products with the underlying theme of collaboration between and coexistence with companies. The need to generate new knowledge returns to *Type 1 Basic Research* and satisfies our own intellectual curiosity. These do not involve a planar rotation of research, but instead use a three-dimensional rotating ring, through which we continue our struggle by never returning to the starting point.

Acknowledgment

Type 2 Basic Research for developing the multiple gene expression detection kit was achieved by the staff of the Cell Dynamics Research Group of the Research Institute of Cell Engineering. Product realization was accomplished by the cooperation between Mr. Masayuki Ryufuku, Ms. Chie Suzuki, Mr. Toshiyuki Takeuchi, and others of Toyo B-Net Co., Ltd., and Mr. Tomomi Asai and Mr. Shigeaki Sakai of Toyobo Co., Ltd. Multicolor spectrum analysis was solved by cooperation between Associate Professor Hidefumi Akiyama of the Institute of Solid State Physics and Mr. Hidehiro Kubota, Mr. Toshiteru Emoto, Mr. Shuji Sekiguchi, and others of ATTO Corp. The research was funded by grants from the NEDO Intracellular Dynamism Analysis Project.

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Postscript

After writing this paper, we received the wonderful news that Dr. Osamu Shimomura won the Nobel Prize in Chemistry for 2008 for the discovery of green fluorescent protein (GFP). Dr. Shimomura discovered the luminescent protein aequorin and GFP when he was attempting to clarify the luminescence mechanism of crystal jellyfish *Aequorea victoria*. The luminescent protein aequorin was realized as a product of the early stage in finding a reagent to detect calcium. At the time, no one believed that GFP would have practical application other than the fact that it was “pretty” protein. This was because no one believed that protein itself could emit fluorescence. Thirty years after the discovery, Dr. Prasher, who did not share in the Nobel Prize, isolated the genes for GFP in 1992 and discovered that the protein itself emits fluorescence. Dr. Chalfie, who received the Nobel Prize in Chemistry jointly with Dr. Shimomura, applied it to animal cells in 1994, and Dr. Tsien created GFP with different colors in 1998. Positioning the roles of theses researchers, Dr. Shimomura and Dr. Prasher engaged in *Type 1 Basic Research*, Dr. Chalfie conducted *Type 2 Basic Research*, and Dr. Tsien conducted *Product Realization Research*. We recognize the importance of *Full Research*, and we appreciate that *Type 2 Basic Research* and *Product Realization Research* have potential to change society.

Dr Ohmiya maintains contact with Dr. Shimomura and can consult him about the research. Dr. Shimomura often points out the importance of *Type 1 Basic Research* and urges those who engage in *Type 2 Basic Research* to continue studying the unsolved luminescent mechanism. He is aware that clarifying the luminescence mechanism may lead to new applications. For example, he points out that copper ion may be necessary for luminescence in certain luminescent shellfish and that a new metal ion sensor may become possible if this luminescent mechanism is clarified. *Type 2 Basic Research* does not evolve without *Type 1 Basic Research*. Conversely, *Type 2 Basic Research* and Product Realization Research are necessary to fully understand and apply *Type 1 Basic Research*. Dr. Shimomura is aware of these relationships.

This year’s Nobel Prize in Chemistry reminds us of the importance of the full range of research activities. Our research group wishes to respond to Dr. Shimomura’s expectation by working on *Type 2 Basic Research* and *Product Realization Research* on the luminescence of the sea-firefly, on which Dr. Shimomura worked previously, and to engage in *Type 1 Basic Research* for unsolved luminescence phenomenon (Ohmiya).

Authors

Yoshihiro Ohmiya

Completed studies in endocrinology, Gunma University Graduate School of Medicine in 1990. Worked as researcher at Osaka Bioscience Institute, researcher of Original Independent Research "Sakigake 21: Light and Materials" of the Japan Science and Technology Corporation, and associate professor in the Faculty of Education, Shizuoka University. Joined the National Institute of Advanced Industrial Science and Technology in 2001. Currently dispatched as professor of photobiology, Lectures in Advanced Medicine, School of Medicine, Hokkaido University from October 2006. Group leader of Cell Dynamics Research Group, Research Institute of Cell Engineering. Worked partly on NEDO "Development of Hazard Assessment Method Using Cultured Cell." Council member of the Japanese Biochemical Society, person-in-charge of bioluminescence and chemical luminescence, and editor of international academic journal Luminescence. In this paper, worked on overall integration.

Yoshihiro Nakajima
Completed studies in production information science at the Graduate School of Science and Engineering, Saitama University in 1996. Worked as a researcher of Basic Special Science, RIKEN, and researcher of future pioneering at the Japan Society for Promotion of Science (Nara Institute of Science and Technology). Joined the National Institute of Advanced Industrial Science and Technology in 2001. Dispatched to the office of the Council of Science and Technology Policy, Cabinet Office, Government of Japan in 2007. In this paper, worked mainly on luciferase and construction and optimization of the measurement system.

Discussion with Reviewers

1 Patent construction for product realization

Question & comment (Hiroshi Kuriyama)

In chapter 3, you wrote that you "developed a method for dividing and quantifying luminescent color without decreasing light efficiency" in cooperation with Associate Professor Dr. Akiyama of the Institute of Solid State Physics, the University of Tokyo, and I think the content of that method is important point of *Type 2 Basic Research*. Can you please describe it in detail?

Answer (Yoshihiro Ohmiya)

We agree that this was not explained clearly. I added Figure 3 and the following sentence. "In the newly developed technology, luciferase is measured without a filter and with several optical filters to determine the transmission coefficient when various filters are used for each luciferase. The amount of luciferase is calculated from the transmission coefficients after the measurements using the various filters (Figure 3)."

2 Construction of multiple gene expression detection technology

Question & comment (Naoto Kobayashi)

I can understand very well that the authors wrote a clear scenario toward the goal of realizing multiple gene expression detection kit, and engaged in R&D along that scenario. In that sense, I can see the strategic effort toward the practical use of the result of simultaneous detection of three gene expressions for the first time in the world, and I think this paper is highly valuable. On the other hand, I think the synthetic description of "multiple gene expression detection technology using bioluminescence phenomena" that is the central issue of this research is lacking. Although the details of this technology have been published in separate papers, I think you should describe the details on how you were able to construct the multiple gene expression detection technology.

Answer (Yoshihiro Ohmiya)

The multiple gene expression detection kit that we developed is a system for analyzing simultaneously the expression of several genes. The kit comprises vectors of the tricolor luciferase gene to which a transcription regulation region can be inserted. The user extracts the transcription regulation region of the gene of interest using a particular method and inserts it into the vector provided with the kit. Transducing this vector into the cell and adding a particular chemical stimulates the expression of the target gene in the cell, and the amount of luciferase synthesized changes. The cell is crushed, the luciferin solution optimized for that measurement condition is added, the amount of luminescence for each color is measured, and the change in the gene expression pattern is assessed. Our patent covers the concept of analyzing the expression of three genes with three colors and tricolor luciferase. The optimized luciferin solution is covered by a corporate patent. The technological method for synthesizing the multiple gene expression detection technology was added to Section 4.

3 Improvements in optical metrology technology

Question & comment (Naoto Kobayashi)

I would like to ask about the optical metrology technology. You mentioned that you used a luminometer equipped with an optical filter for optical measurements. Are the technologies including measurement sensitivity, range, and SN performance complete? Please mention any improvements for the future optical metrology technology.

Answer (Yoshihiro Ohmiya)

Use of optical metrology technology is mandatory for the diffusion of our technology. From that perspective, we have added the following sentences. "In the future, compatibility of data obtained from the simultaneous measurement of several samples will become important in toxicity assessment, and we are developing a new optical measurement device with high sensitivity and standardized optical metrology. Incorporation of new optical metrology technology will be very important for expanding the use of the multiple gene expression detection kit."

4 Future development as an alternative to animal experimentation

Question & comment (Naoto Kobayashi)

The result of this research is greatly significant for the practical application, and I think you had better explain the future developments including whether this method can actually be used as an alternative to the animal experiment, or whether the observation using this method to observe the intracellular dynamics represents the actual *in vivo* reactions.

Answer (Yoshihiro Ohmiya)

We recognize that the technology for obtaining biological information using light is not all-powerful and that any errors in reading the light would lead to the wrong conclusion. When we say that biological information is obtained through light, we mean that the information is the shadow produced by light, so we cannot obtain useful information unless we can measure light accurately. This point has been added to the text along with the following sentences. "We expect that increasing the reliability of the biological information obtained at the cellular level will provide alternatives to animal experimentation. It may also be possible to develop technology "to protect health with light." Looking at several aspects of biological information simultaneously provides a view of the cellular dynamism through light, which may provide new information about life "to support health with light."

5 Improvement of precision

Question & comment (Naoto Kobayashi)

I understand that the “method for detecting the expression of three genes simultaneously using bioluminescence phenomena” is extremely effective. For the improvement of precision in the future, will you need more than three colors? Or do you think three colors are sufficient as a basic method?

Answer (Yoshihiro Ohmiya)

In terms of improving accuracy, I think it is important to perform simultaneous analysis with three or more colors. However, the light that can be made from firefly is green to red light with luminescence peaks at 540-430 nm. Human eyes can detect subtle differences in color, whereas a camera has only a limited ability to distinguish faint light. Because the light of bioluminescence is broad light, we have chosen a detection method that is applicable to broad light with 30 nm separation, and three colors is the limit at present. This points to the need for further *Type 2 Basic Research* to develop a new metrological system to broaden the applications. In addition, in gene expression analysis with more than three colors, the method for gene transduction into cells will become an important issue. Thus, we expect that this *Type 2 Basic Research* will lead to further *Type 2 Basic Research*.

Development of clay-based-film

— A *Full Research* scenario from a viewpoint of encounter —

Takeo Ebina

[Translation from *Synthesiology*, Vol.1, No.4, p.267-275 (2008)]

An example of *Full Research* in the development of clay-based-film is introduced. Clay is eco-friendly, and it exists abundantly in Japan. Clay-based-films can be used as heat resistant gas barrier materials that in turn contribute to the development of a sustainable industry. Details of the technical development, public information, intellectual property, and technology transfer occurring during the process from the initial invention to practical application are discussed. At the same time, the interactions of people and research groups related to the development process are analyzed. In addition, the effectiveness of a consortium is discussed in the context of an innovation model based on development integration.

Keywords : Clay, *Full Research*, *Type 1 Basic Research*, *Type 2 Basic Research*, practical use research, encounter, consortium

1 Film made from clay

Most conventional gas barrier films were manufactured using plastics. Their gas barrier performances were not perfect, and the researches were conducted for "clay-plastic nanocomposite material" in which clay and other materials were added as fillers to enhance heat resistance and gas barrier property. Gas barrier performance was improved noticeably by adding small amount of clay. From "reverse ideation" that gas barrier property and heat resistance may increase dramatically if the clay used as filler was used as main material rather than as additive, in 2003 we started the development of heat resistant gas barrier film composed of clay[1]-[3].

Clay crystal is thin plate about 1 nm (1/1,000,000 millimeter) thick. "Claist" is a clay film formed to thickness enough to be handled by layering several tens of thousands of thin crystals. Claist can be bent and has high gas barrier property against oxygen and hydrogen gases under high temperature condition. It is made by cast method, which is a simple method where clay dispersed in liquid is dried in a tray, and the dried precipitate is peeled off. We conducted film preparation using various types of clays to study their film-formability. We found that clay called "smectite" that readily formed water gel had excellent film-formability[4]. About 30 to 70 % of smectite is naturally contained in mineral called bentonite. Bentonite itself is used as moulding agent of casting sand, drilling mud in construction sites, and impermeable layer of dam and waste disposal site. About 450,000 ton/year are produced in Japan, and this is almost equivalent to the production volume of virgin PET by Japanese companies. More than half of this amount is produced from mines in the Tohoku region. Separation and refining of smectite from bentonite is done by sedimentation, which is a method where the material is dispersed in water and the fraction that does not settle is heated and dried.

That the clay forms film is not a new finding for those of us who study clay. When analyzing crystal structure of clay by x-ray diffraction, the usual method is to use oriented sample where clay-dispersed liquid is cast on glass plate[5]. Since the clay film cannot be peeled off the glass plate, it is not self-standing film[Term 1]. Professor Ernest Hauser of MIT reported self-standing clay film in 1938[6]. Intended use was wrapping material or alternative to paper. Although the potential of clay as film material was indicated, it seemed not to be commercialized as product. It may be because no functional or economic advantage could be discerned against competitive material paper.

Seventy years have passed, and there are now many products that require gas barrier such as food packages and electric products. Moreover, special needs arose for containing hydrogen at high pressure while maintaining lightweight as system to be installed in vehicles such as rockets, aircrafts, hydrogen vehicles, and fuel cell vehicles. In petrochemical plants, leak must be controlled as much as possible to reduce volatile organic compound gases. New materials using clay were deployed to meet such new demands.

2 Invention of clay-based film and establishment of the concept

2.1 Discovery of gas barrier property of clay-based film

The Author was initially studying clay compaction to assess artificial barrier in waste disposal sites[7]. Permeation of water through clay compaction was extremely slow, and it required long time for measurement. In some cases, maximum 500 days were necessary for measurement. To complete the measurements in short time, we devised ways of thinning

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the compaction that was about 16 mm thick. Although measurement time was shortened using the thin material, the uniformity of the film greatly affected the measurement precision. We repeatedly formed film on filter paper to achieve uniform thickness. At this stage, we were working on clay film with focus on its function as water barrier. The finding of this project was reported but did not attract much attention.

AIST Tohoku was established in 2001, and research unit “Research Center for Compact Chemical Process” was established in 2004. There, I presented this film to my supervisor who commented that it could be used as sealing material of micro-reactor using hydrogen gas, and I started research of clay film as gas barrier material.

Self-standing ceramic film had numerous small cracks through which air molecules could pass. Therefore, high gas barrier property could not be expected, and ceramic film has not been considered for gas barrier. Clay film certainly showed high barrier property against water, but that was because clay absorbed water, expanded, and filled the cracks. Therefore, people who made ceramic films never considered using them as gas barrier material. The idea that clay film might be used as gas barrier material was probably gained from the fact that the appearance resembled the sealing material called Teflon tape. Teflon tape was actually used as seal in micro-reactor, but it can not withstand over 250 ºC. Efforts were continued on clay film to improve strength and flexibility like Teflon tape, and to reduce pores that resulted from air bubbles. The idea for use was born first from the appearance of the material, and then expanded through human five senses such as sight and touch.

2.2 Demonstration of gas barrier property of clay-based film

While clay film lacked sufficient strength initially, improvements were repeated based on discussions with my supervisor, and in few months, we had a material that could be used as seal in micro-reactor. In the micro-reactor trial, hydrogen had to be sealed off up to about 300 ºC, and no conventional sealing material could achieve this condition. Upon assessment, sealing property achieved to be good. In principle, it was thought that clay did not hinder permeation of hydrogen, but it was unknown whether the sealing property against hydrogen would be lost by addition of binder. Later, using the tuturous model[8], it was found that high gas barrier property was obtained when clay was excessive. Specifically, gas barrier property of 1,000 times higher than the binder material could be obtained when the clay weight ratio was 94 %. The high barrier property according to the principle was experimentally demonstrated, and the basic concept of clay film was established[9].

3 Application development of clay-based film

3.1 Analysis of demand

Press release for clay-based film was issued on August 11, 2004. The highlight of the release was heat resistance of maximum 1000 ºC and gas barrier property lower than detection limit. We also frequently participated in exhibitions, and also wrote several technical reports in journals.

As result of concentrated publicity in short time period, there were over 300 inquiries, and we accepted technological consultations with about 150 companies. It was found that this material held potential for diverse usages including heat resistant flexible film, display film[10][11], graphite composite material, electromagnetic shielding material, condenser seal, packaging material (high barrier paper container, high barrier flexible package), and hydrogen sealing material.

3.2 Enhancement of intellectual property rights

Looking at the timing of disclosure of patent application, presentation at academic societies, and publication in journals, we took measures to enhance the patent in the council that discuss patent enhancement among the Intellectual Property Department, AIST Innovations, and research units. Specifically, preceded technological surveys were conducted in August 2004, December 2004, and February 2005, and enhancements of intellectual property were intensively conducted by Intellectual Property Strategic Enhancement Team (organization composed of smaller number of people than Patent Enhancement Council, to enhance certain intellectual property) in December 2004, June 2005, March 2006, May 2006, and September 2006. Selections were made on fields and R&D topics that should be developed internally within AIST, and fields and R&D topics that should be developed jointly with private corporations. Specifically, we decided to concentrate our application on material patent, part of manufacturing patent, and part of application patent that could be developed in AIST. Patents that were applied for application included flexible substrate, solar cell, and materials for fuel cell. These were areas to which clay-based films were provided within AIST and good results were obtained in preliminary tests. Of course, some did not go well and applications were not submitted. During this period, about 40 patent applications were applied. Attainment of sufficient quality and quantity of the intellectual properties and application for patent individually by AIST for this technology started in 2003 and reached peak in 2004-5.

3.3 Policies for technological transfer and joint research

Technological transfer was conducted through research sample provision contract and research information disclosure contract. There were about 70 former cases and about 20 latter cases. In technological transfer, we worked to accumulate sufficient manufacturing know-how and to communicate this know-how accurately and in detail. In research information disclosure, we actively accepted observation inside the lab as well as disclosure of know-how book and presentation of undisclosed patents. Disclosure of undisclosed patents and know-how books were not very detailed, and the companies often could not reproduce the results completely with that information only. As result of lab observation, most companies were able to successfully create film with quality equivalent or higher than the AIST sample, and this style of research information disclosure proved to be effective in transferring technology.

In 2004, we commenced joint research by different usages based on research information disclosure contract. Such joint researches increased around 2005, and the development stage of clay-based film gradually shifted to Applications Research. Even in case the film would be used in same product, if division could be made between upstream and downstream, we made it possible to start joint research. It was thought that as the R&D stage progressed, vertical collaboration from upstream to downstream companies would accelerate R&D. At the same time, there was possibility of conflict of interests, and it was necessary to take measures to maintain friendly development relationship. The employees of AIST could not be involved in details due to nondisclosure obligation, and it was useful to have a consortium composed of major research members. The Research Center for Compact Chemical Process is an organizer of Green Process Consortium (GIC), and supports horizontal and vertical collaborations among companies.

The number of joint patent applications with companies increased from 2006, and in 2007, 80 % of all patents filed were joint applications. One characteristic of clay-based film development is that there are more than 10 companies with which joint applications are filed.

4 Development of asbestos-substitute gasket

Among the companies from which we received technological consultation, we decided to develop sealing material for use under high-temperature condition with Company J in the Project to Support Regional Small & Medium Companies, Ministry of Economy, Trade, and Industry in FY 2005. Based on the results obtained, we developed gasket product with heat resistance, durability, and chemical resistance superior to those of existing non-asbestos products, as well has excellent manageability equivalent to asbestos products, by compositing expanded graphite and heat-resistant clay-based film, as project of New Energy and Industrial Technology Development Organization (NEDO). The newly developed gasket can be used widely in chemical plants such as oil refinery and thermal power plant.

4.1 Problems in conventional asbestos-substitute gasket

In many chemical industries, gaskets are used to prevent leakage of liquid or gas in the pipe joints in the production process under high temperature condition. Asbestos products were used widely in high temperature area. While immediate measures had to be taken against asbestos health hazard, development of alternatives was in progress and assessment of safety and reliability were not sufficient. Expanded graphite gasket had many advantages such as excellent sealing, long-term storage, and ease of handling, and was highly regarded as non-asbestos product. However, bonding among graphite powders was not strong, and there were problems such as “powdering” where powders flaked off from the product surface, and “adhesion” where the graphite burned on to the metal surface where the gasket came into contact. Moreover, in oxygen atmosphere and temperature of 400 ºC or higher, oxidation and deterioration progressed and the gasket wasted away, thus loosing sealing property.

4.2 Scenario setting

4.2.1 Improvement of heat resistance of gasket by compositing expanded graphite and clay

It is difficult to solve the phenomenon where graphite burns in presence of oxygen at 400 ºC or higher. However, clay is oxide and has high heat resistance. Clay used in clay-based film is stable to about 600 ºC, and we considered improving the overall heat resistance by mixing and compositing clay. Since clay-based film has high impermeability against oxygen, we thought it was possible to lengthen the lifespan of gasket by slowing down transfer of oxygen by coating with clay-based film.

4.2.2 Prevention of powdering and burn-on by addition of clay layer

Powdering of expanded graphite could be solved by applying an uniform coat of clay-based film on the surface as in the current fluororesin products. Burn-on was thought preventable by isolating metal surface of the flange from graphite by clay-based film coating.

4.2.3 Improvement of sealing performance by flattening the gasket surface

It was possible to reduce the amount of liquid that leaked between the expanded graphite gasket and metal flange by flattening the surface of clay-based film used to coat the gasket.

4.3 Elemental technology

4.3.1 Nanocompositing technology

Clay has higher heat resistance than plastic. Also it expresses gas barrier property by fine preparation. However, there were three major issues in using clay film as gas barrier material. One was existence of cracks. It was not easy to completely remove small cracks through which gas molecules could pass even though clay-only film appeared uniform. In gas barrier material, just one crack may cause performance deterioration. Second was low mechanical strength. Although clay film could be bent, it was not flexible like plastic. The film was weak, and once a crack formed it could be easily broken. Third was weakness against water. As mentioned earlier, the condition of clay to be used in self-standing film makes good aqueous dispersion, but this also meant that clay film dissolved readily in water. It had problem of poor steam barrier property that was often demanded of gas barrier material. Unless these three issues were cleared, there was no future as general-use gas barrier material. It seems that fairly large number of engineers gave up because they were unable to solve these issues.

To solve these issues, nanocomposite technology was applied. Clay raw material and organic compound that would act as binder were uniformly mixed at microscopic level to form the film. In many cases, dispersal technology involving pretreatment using the charged state of clay surface was used. By nanocompositing, it was possible to remove the crack on the clay film and to improve mechanical strength and water resistance. While in ordinary nanocompositing, small amount of inorganic material was introduced into organic material, in case of clay-based film, small amounts of organic material were present in the clay, and the composition was opposite.

4.3.2 Clay-based film manufacturing technology

Thousands of sheets were created to obtain the best film-forming method. Ultimately, we made clay film every day for five years, and were able to accumulate the know-how of film forming. As result, we could create clay film of 10 μm thickness with good reproducibility. At the same time we investigated the coating method, and learned that dip coating, spray casting, cast method, and bar coating method could be applied. During the aforementioned research sample transfer contract, we spent effort on quality control even at lab level. The samples were carefully created, reproducibility checked, and main property values were obtained as much as possible by subcontracting to external lab to supply property value chart. We established clear specification of film quality before shipment. Specifically, they included size, level of unevenness of thickness, and level of unevenness where clumps could be observed by naked eyes. Such quality control was useful in accumulating manufacturing know-how of clay-based film.

4.3.3 Clay library

In the process of looking for appropriate clay for film, about 130 clay samples were collected from Japan and overseas. These were natural or synthetic clay, and most were available commercially. It included inexpensive clay samples without purification. The data collection of assessment of film-forming property of these samples is in progress, and clay suitable for clay/expanded graphite composite was selected from this library.

4.4 Integration process

4.4.1 Selection of coating method with good adhesivity to expanded graphite

Heat resistance of at least 400 ºC was required for the gasket. Therefore, organic adhesive agent could not be used. Initially, pressing method was used. Although certain adhesivity was obtained, there was problem of air entering between expanded graphite and clay film. Dip coating had advantage of prepareing coating layer to form on the sides. Upon trials, clay film of about 20 μm thickness could be formed on the expanded graphite surface. Water was used as solvent.

4.4.2 Selection of appropriate combination from various raw materials (clay and additive, clay blend)

We worked on selecting clays from the clay library with excellent adhesivity to the surface of expanded graphite. Since transparency was not demanded in this clay-based film, screening was done mainly for natural clay due to cost concerns. As result, it was found that film forming and adhesive properties were excellent in clays that contained mineral called smectite. Epoxy, phenol, and polyamide resins were selected as additives, and optimal material among them was selected in the final specification. For heat resistance, the amount of additive was minimized enough to maintain mechanical strength. The solid-liquid ratio of coating paste was increased to shorten duration required for drying, and clay blend was investigated to create film with excellent fabrication property. Coating film with excellent film property as well as fabrication property was made using clays with different types.

4.4.3 Feedback system for utilizing the assessment result of element test and actual plant trial

Element test was conducted by gasket manufacturer Company J, and good results were obtained for sealing, sticking, and manageability assessments.

In the product development process, we were able to obtain cooperation of Company M, a user company and a member of GIC. Testing was done by installing a bypass in actual petrochemical plant even though the gasket had no previous record of performance. This vertical collaboration effort was deployed under NEDO Urgent Asbestos-Substitute Development Project, and feedback system was established where element test and assessments in actual plant test could be utilized for improvements. Basic data on safety and reliability was obtained and asbestos-substitute gasket was commercialized for use under high temperature condition in 2007[12]-[14]. A gasket factory constructed and started the operation in Osaka in September 2007, and the product is used in about 40 plants throughout Japan as of July 2008. Asbestos-free plants were realized with the introduction of this gasket. This result was acclaimed highly, and the gasket won the Exellent Award of the 2nd Monodukuri Prize in 2007.

The primary reason this product development was achieved in short time was due to precise idea from the president of Company J who thought the problem of surface powdering and burn-on that were disadvantages of conventional expanded graphite gasket could be solved by potential technology of clay-based film. The second reason was smooth technological transfer; third was relentless effort of the engineers at Company J; fourth was cooperation and decisions of users obtained through GIC; fifth was quick managerial decision of Company J and practice of technological sales throughout Japan; sixth was close network between NEDO and AIST; and seventh reason was the fact that production was possible by single company.

External factor was the goal for total abolishment of asbestos products by 2008, and the market demanded substitute products. This newly developed product can replace about 70 % of the asbestos gasket products, and we plan to conduct further performance assessment tests, as well as improve long-term reliability, and plan to expand into automobile industry and electric industry as well as chemical plant industry. At the same time, we are developing products for use in higher temperature.

As AIST's internal project, original R&D project to produce the patents held by AIST were conducted in FY2006 and 2007. Also, several grant-based joint research are in progress, and we are steadily moving on toward second and third successful developments.

Creating actual cases of product development and accumulating basic and production technologies at this stage are keys to success of development. Mass-produced, small profit products must have balance between supplier's production technology and user's product development research, and certain time is necessary for shaping of the market. On the other hand, product development of gasket was done by single company, and development was accomplished in short time without waiting for formation of corporate collaboration. If there are cases of developments, the desire for product development of researchers, engineers, and managers in other fields increases, and this is another reason we think creating successful cases is very important.

5 Encounter and research development in *Full Research*

Here I shall analyze the encounters in *Full Research* using as example the development process of clay-based film.

Specifically, I shall categorize the encounters in the processes of development of invention and progression to *Full Research*.

In *Type 1 Basic Research* stage, about 70 years have passed since humans invented clay film and about five years since the Author started research on clay-based film. During that time, changes took place in the types of industrial products and demands in performance of materials, and new potentials arose from the old seed technology. The manufacturing know-hows of clay film were accumulated from the basic research of barrier for disposal sites of high-level radioactive wastes and industrial wastes. These basic technologies had been achieved to some level and were available as papers and reports. With the establishment of new research unit, researchers of different backgrounds were able to encounter, and development of sealing material was requested from the Author's supervisor who faced the issue of sealing in micro-reactor[3][15]. There were two points in this encounter. First was the match between potential and demand; second was proposal without preconception.

For the first point, detailed explanation using Figure 1 is provided. For a breakthrough such as discovery or invention to happen through encounter of individuals or groups A and B, at least A should have the desire to seek solution to the problem. Specifically, to achieve highly specialized R&D I, A strongly demands technological development element X that A does not have. B has technology α that is result of R&D II, and α can contribute to X. Contribution of α to X may be close to 100 %, or it may be about 50 %. When A and B encounters, technology for α is presented by B to A. Even when the contribution of X to α is not particularly high, strong desire will push actively search for potential, and potential of contribution of α to X will be found. If the contribution of α to X is high, since the time and resource (people, budget, and facility) for R&D I are available, invention and discovery are achieved in short time. If contribution of α to X is not high, it is necessary to raise the contribution of X by further developing α by feedback to B from A. In this case, cooperative agreement between A and B is necessary. Cooperation may assume the form of joint research III. This stage may be done within AIST or simply as request or order within the research group. At this time, if research II is completed for B's technology α and utilization is being sought, thinking of both parties can match and agreement can be readily obtained. In case research II is in progress, the information may not be disclosed completely and cooperation may be limited.

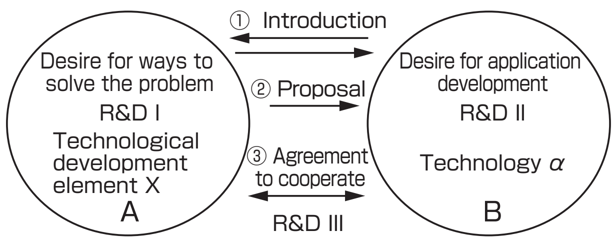

Fig. 1 Encounter in *Full Research* and mutual relationship.

Table 1. Style of encounter in gasket development.

Stage	A	B	X	α	R&D I	R&D II	R&D III	Content of Research	Opportunity for encounter
Type 1 Basic Research	Supervisor	Clay researcher	Hydrogen gas sealing material for micro-reactor	Uniform film using clay-based film	Micro-reactor development	Water barrier research using clay compact	Grant within AIST	Confirmation of high barrier performance, investigation of high barrier performance mechanism	Start-up of new unit
Type 2 Basic Research	Company J	AIST research unit	Improvement of heat resistance of gasket	Heat resistant gas sealing material	Grant within AIST		Support Program for Regional Small and Middle Company, MITI	Development of clay expanded graphite gasket and packing	Technological consultation
Product Development Research	User Company M	Company J + AIST research unit	Asbestos-substitute gasket product	High-performance gasket	Support Program for Regional Small and Middle Company, MITI		NEDO Urgent Asbestos-Substitute Development Project	Development of non-asbestos gasket	Consortium (GIC)
Innovation	User Company Group	Company J + AIST research unit	High-performance gas sealing product	Development technology for high-performance gas sealing product	NEDO Urgent Asbestos-Substitute Development Project		NEDO R&D Project for Industrializing University-Originated Technology	Development of general-use high-performance gas sealing product, data collection for standardization	Product advertisement, etc.

Here, flexible search of potential is expected from A. For example, for technology α, one must use imagination such as thinking about combining with other technologies. It is also necessary to flexibly change the research plan. Next, appropriate explanation for originality is expected from B. Since A does not know technology α very well, it may understand originality wrongly. For the same reason, it is necessary to provide objective advice on whether employment of α is the most appropriate choice among several technologies.

Table 1 is the application of this model to the development of the clay-based film. In *Type 1 Basic Research* stage, clay researcher is B and supervisor is A. In *Type 2 Basic Research* stage, Research Center for Compact Chemical Process is B and Company J is A. In both cases, the encounter is almost ideal. Moreover, in the Product Development Research stage, Research Center for Compact Chemical Process and Company J are B and user Company M is A. The points of this model are: it can be used for analysis for each stage of *Full Research*; and there is optimal timing of encounter. In the process of progressing from *Type 1 Basic Research* to Product Development Research, encounters occur from inside of the organization and spread to outside. Specifically, they spread from highly specialized people who mainly study clay-based film to people who are less specialized, external researchers who have registered ID issued by AIST and to manufacturers and sales people who do not have registered ID issued by AIST.

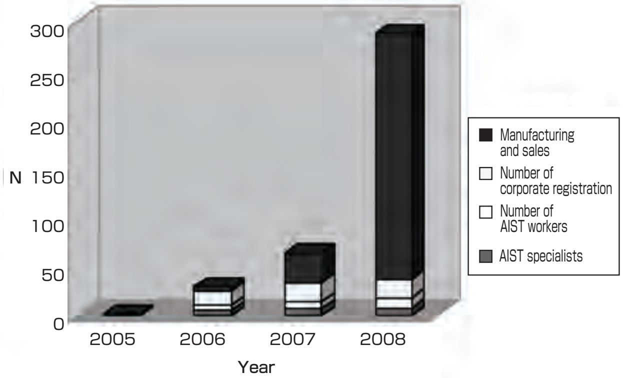

Fig. 2 Expansion of people in clay-based film research (N is number of people involved).

Figure 2 shows the spread of people involved in clay-based film. One of the indexes of evolution of innovation is the fact that the number of people involved increases and population who can benefit as users increases.

6 Integrated development model

Above was the discussion of product development for clay-based film. Product Development Research for clay-based film is divided by products, and these are done with different companies. Also, in some cases, the relationships are upstream and downstream. Specifically, they include companies that produce clay, companies that manufacture clay-based film, and companies that manufacture products using clay-based film. Also, along with vertical relationship as described above, there are companies that share common technological elements, though for different products. For example, they include high water resistance, improvement of transparency of film, or high gas barrier property. To obtain result through balanced development with involvement of all the companies, individual development should shift to accelerated development through information exchange. Since content and progress of joint research with other companies cannot be communicated due to nondisclosure agreement, method for enhancing corporate collaboration through mediation by AIST must be devised, and we are trying to promote collaboration by gradually lowering the walls between the companies using aforementioned GIC. The year 2008, when a industrialization of the product is achieved are produced and joint patents are disclosed, is an appropriate time to start mergeing technologies. In the above model of encounter, the preceding company should be B and the following company should be A in terms of establishment of original technology. AIST does not need to be directly involved in such collaborations.

The multiple development style based on collaboration is called "integrated development," and is compared to the case of "individual development" where no particular collaboration is done. Figure 3 shows the case where product development A, B, and C of clay-based film are done by integrated development and individual development in solid line and dashed line, respectively. In the case of individual development, development of entire clay-based film is equal to the sum of each product A, B, and C (II). The shift is made to integrated development at time T when product development A is achieved. By providing part of technology and know-how accumulated during the development process of product A, development of product B is accelerated. The technology and know-how accumulated during the development of A and B are utilized in the development of product C. Product development D which could have been only conceived in integrated development may be born. As result, overall development of clay-based film is accelerated (I). In bad scenario, collaboration among corporations may fail, each other's technology cannot be used, and product development may slow down compared to single development (III). In this case, product development may be withheld until the expiration of patent (III).

The case corresponding to D that was born by integrated development includes the development of hydrogen gas barrier material for high-pressure hydrogen gas container[16][17]. This is material where clay-based film is sandwiched between plastic sheets reinforced by carbon fiber, and is in progress with cooperation from manufacturers with potential technology for carbon and clay-based film composite material.

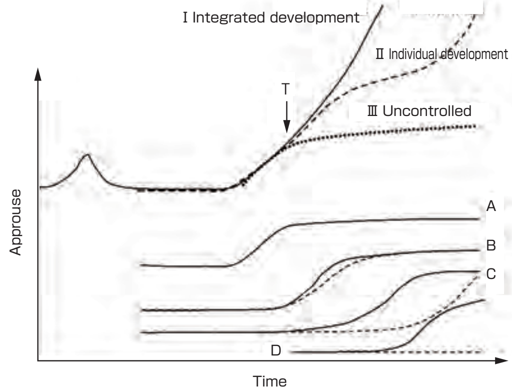

Fig. 3 Concept of integrated development.
A-D are individual product development, solid line is integrated development, and dashed line is individual research.

7 Claist Association

To realize integrated development, we established the Claist Association in August 2008. As mentioned above, GIC, which was a research group for industry-academia-government collaboration at AIST Tohoku, played a major role in the development of Claist, and the Claist Association was created as a subcommittee. As of July 2008, about 30 private companies are members, and we plan to promote practical application of clay-based film while maintaining close collaboration among companies (Fig. 4).

Specific activities include provision of latest technological trends such as patents and papers, management of clay library, operation of Association encounter, and provision of common core technology. Although there are some AIST researchers among the administrator, most are corporate members. That is because it is important to operate the association so it will not interfere with integrated development.

8 Summary

For development of clay-based film, strategic maintenance of intellectual property (quality and quantity) was accomplished through combination of publicity, intellectual property, technological element research, and technology transfer while being strongly schedule-conscious. Manufacturing know-how of clay-based film and roadmap to development were also established.

Encounters at each stage from *Type 1 Basic Research* to Product Development Research were important. Such encounters can be generated by fusion of research groups, publicity, and establishment of a consortium. To incorporate

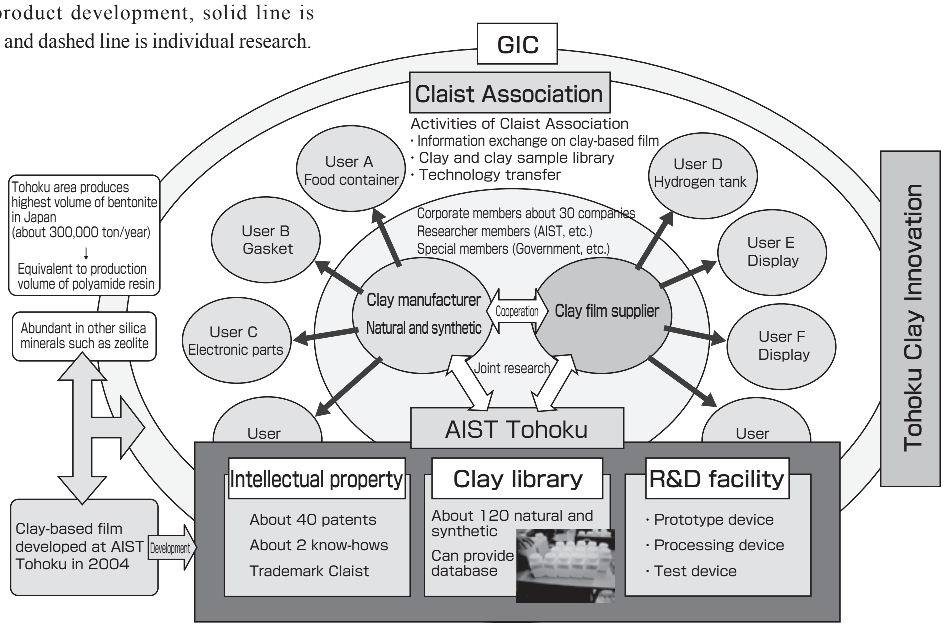

Fig. 4 Mutual relationship between Claist Association and members

these activities strategically into the development process is effective in accelerating *Full Research*. In the case of clay-based film, asbestos-substitute gasket was produced as development case. Through controlled information disclosure, AIST became the center of technology, intellectual property, and information, and took initiative of development. This also means that it has to act as mediator of companies. Consortium activity is an effective way of efficiently mediating and integrating individual product developments.

Finally, by presenting the story chronologically, the latter half of the paper emphasized Product Development Research. However, it is important not to neglect basic research that must be undertaken by public research institution, and it is necessary to maintain manpower for basic research. Currently, we are engaging in basic research to clarify the details of film-forming mechanism of clay and to obtain film flexibility. As result, we are about to generate synthetic clay with film-forming property almost equivalent to natural clay.

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Terminology

Term 1. Self-standing film: Film that can be handled without supporting layer, unlike film that is used to coat a member.

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Discussions with reviewers

1 Overall structure of the paper

Comment (Hiroshi Tateishi)

Since you discussed *Full Research* thoroughly, the focus has become unclear, and the part on *Type 2 Basic Research*, which is the main point of *Synthesiology*, is insufficient. Particularly, I think the first half is merely an "explanation" of the development of Claist as a whole. Since I think the focus of the paper is "the practical application of Claist," I think you should reorganize the structure of the paper.

For example, if "the dream of research" is "the realization of clay-based film and the specific example is the development of an asbestos-substitute gasket," and "the social value of research" is "response to new demand through heat-resistant gas barrier film," I think the corresponding scenario setting, description of elemental technologies, and description of the integration process are necessary. For the paper, you need not to describe all components chronologically, and I think you should sort, select, and discard a bit more.

Answer (Takeo Ebina)

As result of trying to include explanation for all elements of *Full Research*, certainly, the focus has become unclear. I reviewed the structure for the points you indicated. Since "dream of research" is "development of clay-based film and specific example is development of asbestos-substitute gasket," and the "social value of research" is "response to new demand through heat-resistant gas barrier film," I included the corresponding scenario setting, description of elemental technology, and description of the integration process.

2 Addition of process leading to reverse ideation

Comment (Hiroshi Tateishi)

I think the impact will be stronger if you describe how you achieved "reverse ideation" in the discovery of gas barrier property of clay film. In the current explanation, it seems like you just happened to think of it.

Answer (Takeo Ebina)

The discussion on how "reverse ideation" lead to the invention was insufficient. For this point, I described how effort was spent in routine research to achieve invention that other researchers cannot easily attain alone.

3 Mutual relationship of encounter in *Full Research*

Question (Kazuo Igarashi)

In Fig. 1, A is desire for potential and B is desire for application. The two are different phrases but seem to talk about the same thing. Are there differences? Also, in relationship between A and B, it seems that A always takes the initiative, but is this correct understanding?

Answer (Takeo Ebina)

As you mentioned, it does seem that A's desire for potential and B's desire for application seem to talk about same thing. Reconsidering this point, A is desire for ways to solve the problem. Since research is progressing chronologically in B, it can be called desire for application of research result obtained, rather than desire for problem solving. For this point, I changed A to desire for ways to solve the problem.

It seems that in the relationship between A and B, A is always taking the initiative. This description is forced categorization, and I think discussion is necessary of which cases fit this pattern. Yet there are two reasons for setting different positions for A and B. First, I thought this analysis might be useful in consciously creating opportunity for encounter. By making decision on whether one's status is A or B, there will be advantage in how to seek partners efficiently and how to set policy for fruitful encounter. Second, although it is possible to set A and B at same position, in this case discussion from each position cannot be done in the encounter, so it was necessary to give A and B some characteristic. The two who seem to be in same position can be given characteristic as A and B as the content of technology becomes more refined. For example, in one project, there are technological development factors X1 and X2, and technological development element X1 was A's position and factor X2 may be B's position. The two is even in total. Also, I think there are cases where one of them will take initiative in short time, and then shift to joint development.

4 Organization of concept of integrated development

Question (Kazuo Igarashi)

In Fig. 3, it is explained that D can be born as result of integrated development, but seen chronologically, the line starts quite earlier than point T. What is the reason? What does the flat line mean?

In the caption, it is written than D is also individual development, but does this mean thing born from integrated development is developed individually?

Answer (Takeo Ebina)

Figure 3 can be interpreted wrongly as it, and I think it must be corrected so it could be understood readily. As you indicated, D should have the line starting from point T. Solid line is change in case of I individual research, and if this is II integrated research, breakthrough will occur in earlier stage from B to D. I decided to add this line. The cases where the product developments of A, B, C, and D for clay-based film are developed integrated and when they are done individually are represented by solid and dashed lines, respectively.

Development of a risk assessment system for soil contamination and the application to the social system

— Processes in *Synthesiology* for practicing an advanced environmental risk management —

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In developing risk assessment technology for soil contamination, it is essential to carry out a wide range of fundamental research and to synthesize the results into a single system. We have carried out an integrated research project for a comprehensive risk assessment system; this project includes fundamental knowledge, database formulation, commercialization of technology, and the introduction of the research results to a social system. In this paper, we present a scenario in *Synthesiology*, the integration of each part of the research results, and discuss the spiral processes involved in the implementation of risk assessment.

Keywords **:** Soil contamination, groundwater pollution, risk assessment system, risk management

1 Introduction

The paradigm to manage soil environmental regulations in Japan has been shifted from governmental management into a self-governance framework, which is a risk-based approach on the basis of site-specific situations and environmental conditions at contaminated sites. The advanced paradigm for the governance framework was oriented from the methodology of exposure and risk assessment. Environmental media, such as soil and groundwater, compared with other media of air and water, exhibit the features of the limitation of exposure of contaminants from soils and the ease of controlling exposure to humans. Thus the risk assessment should include the mechanism of the exposure scenario specified at each site and under various geological conditions. In developing the risk assessment system for soil contamination, it is important to consider the variation of soil properties and groundwater flow characteristics relevant to the geo-environment.

This study focuses on the development of a comprehensive risk assessment system for the geo-environment that includes various parts of soil, groundwater and sediments. In this research, we designed a program of full and integrated research of geology and environmental sciences in order to combine various technologies, because only conducting individually each research work is inefficient for the completion of risk assessment system. This approach has yielded great success in research and development, and the paradigm has changed to solving the problems of soil contamination by means of the spiral process of risk analysis and management.

In this paper, we present the development of the geo-environment risk assessment system, an outcome of the full research comprising of combination of various research areas, the components of integrated research, disclosure and widespread utilization of the developed system in industrial and social applications, and the trials of applying the output for environmental management and the social system. Furthermore, the advanced scenario in *Synthesiology*, from *Type 2 Basic Research*[Term1] to *Product Realization Research*[Term2], is also discussed to allow for the integration of various technology components.

2 Current situations of soil remedial action and research objective

The number of soil contamination surveys and remediation in Japan has increased very rapidly since the enforcement of the soil contamination countermeasures act in 2003. According to the current record published by the Japanese Ministry of

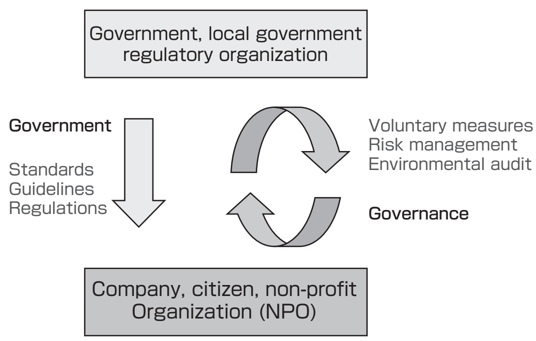

Fig.1 Structure of governance and contribution to environmental improvement.
This shows the difference of social system and approach of environmental management between legal system (government) and voluntary management (governance). The methodology of risk assessment and management is mainly applied in the approach of self-governance.

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the Environment, the official number of registrations by local governments for soil and groundwater remedial action was approximately 1200 in 2006, although the number of self-governance actions at industrial factories was much higher than the official value[1]. As the social background behind this trend, there are some typical situations of increasing environmental awareness and the active land transactions in recent years. Fig.1 illustrates the paradigms of environmental management; one of them is merely governmental action, such as environmental regulation, and the other is the governance approach by a loop-like various stakeholders including industry, citizens and non-governmental organizations. The spiral approach of the participation of all stakeholders is very important for achieving the successful management with multilayered governance[2]. The amendment of the soil contamination countermeasures act has come under review, because a new framework of cooperation between government and self-governance should be made applicable for comprehensive environmental management[3].

According to a survey by the Ministry of Economy, Trade and Industry of Japan, more than 90 percent of soil remedial actions were based on self-governance framework instead of the soil contamination countermeasures act. Those activities have been implemented by land or industrial plant owners in environmental management system, by means of voluntary measures[4]. However, there are no unified requirements on the methodologies of exposure and risk assessment and remediation actions, particularly for voluntary measures. Because of the lack of scientific information on exposure and risk, it is necessary to develop the risk assessment system that can be adopted for Japanese situations of soil and groundwater, from which we might be commonly exposed by environmental contaminants[5].

In taking such social and regulatory situations into account, integrated research work with various technology components has been carried out to complete the geo-environment risk assessment system. The role of governmental contribution in environmental policy and management is very important and is requested by local government and industries. This series of research include all related studies, such as the development of the risk assessment system, geological survey and data acquisition on site, and numerical simulation, as well as the disclosure and widespread utilization of the developed system. One of the significant outcomes is the introduction into social system for industrial location utilization, on the basis of product planning of the risk assessment system, and the development of databases necessary for such assessment. The final goal of this research is to establish the standardization of this risk assessment system for reasonable measures of soil remediation action in Japan. In addition, this system has some advantages, compared with other published risk assessment systems worldwide, such as the consideration of land use classifications, and the types of soil and geological media inherent in Japanese situations[6]. Thus we focus on widespread acceptance and utilization in many countries worldwide, particularly to Asian countries facing serious environmental pollution. Risk mitigation and cost reduction are the great contribution to any sector that uses the developed risk assessment system.

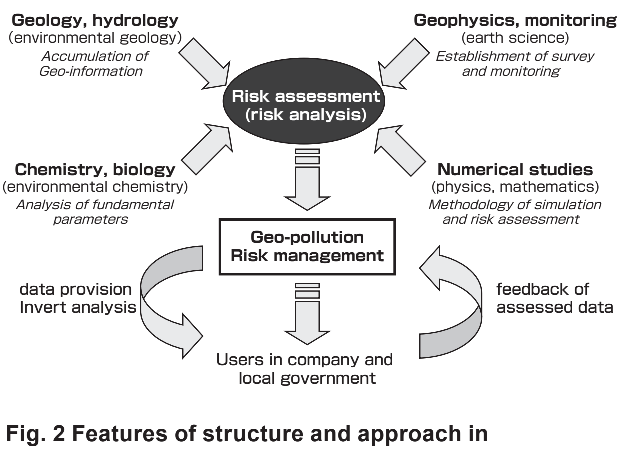

Fig. 2 Features of structure and approach in *Synthesiology* in this research.
This shows the scenario on the integration of technology components needed for competing the development, where a spiral structure is well characterized in developing the system and databases.

3 Significance in *Synthesiology* and research perspective

Fig.2 shows the processes of risk assessment in this research and spiral architectures based on the system development and user feedback. The research project is aimed at completing the risk assessment system, on the basis on of not only the accumulation of technology components, but also the integration with relevant analyses of each technology components. The original concept of introducing processes in *Synthesiology* to obtain the final goal is described below.

The existing environmental laws in Japan normally involve a uniform system of environmental criteria classified under concept of government action. On the other hand, in the current process of risk management, the regulations

Table 1 Basic study areas and technology parts relevant to this research.

Exposure and risk analysis	Risk science (information science)	Risk analysis, Exposure analysis
Exposure scenario and factors	Safety science (toxicology)	
Geological structure database	Geology (urban geology)	Geological survey, Groundwater flow analysis
Soil and groundwater properties database	Environmental geology (hydrology)	
Flow and transport database	Earth and geo-sciences	Geophysical exploration, Environmental monitoring, Chemical analysis on site
Parameters of interaction with soil and organism	Environmental chemistry (chemical, physical analysis)	
Chemical substance database	Physical chemistry (equilibrium and kinetics)	Chemical partitioning, Biology/ecology
Datasets for environmental pollution	Environmental sciences	
Numerical simulation method	Fluid and computational sciences	Numerical analysis, Visualization of results
Formulation and visualization	System engineering	

have shifted to the system of self-governance, as shown in Fig.1. Since the process of self-governance is based on the participation of multiple stakeholders, it would be an effective measure in the environmental management system in the near future, considering the possible industrial structure with sustainability[7]. Thus this research adopted the original scenario of the integration of technology components and the interactive communication, on the basis of a unified index of environmental risk, to obtain the change in the paradigm of environmental policy.

The integration of fundamental studies, such as those in geology, environmental and earth sciences, is essential to achieve the landmark of system development, as listed in the table. In particular, information on soil properties, groundwater aquifers, and geological structure at sites is essential to obtain reliable assessment results. The characteristics of chemical and existence forms of pollutants and the parameters of exposure are also very important in realistic risk assessment. These research parts have features with complementary relationships that should be integrated to a uniform system by means of a common criterion. In the research program we intended to make up of a scientific consistency for data and parameters needed in the risk assessment. Thus we conducted the uniformity of methodologies and research management.

Some basic research, such as the formulation of equations for exposure assessment and the numerical simulation, are principal areas in the development of the risk assessment system. In addition applied research areas, such as the technological parts on geophysical exploration and groundwater hydrology at contaminated sites, are also quite important for the integration. Therefore, we have made some efforts to correlate these research results into the product of geo-resources risk assessment system, synthesized with a unified index of “environmental risk”. The verification of methodologies and parameters used in the system is essential to complete its development. Thus the assessment data is validated with the monitoring results to be obtained through feedback from the users, and experts and professors finally examine the system to validate it for public application.

4 Study on technology components and methodologies

The technologies needed for the development of the geo-environmental risk assessment system cover a broad range of components. There are various elemental sciences and technologies, such as geological survey, contamination assessment, analytical and simulation methods, monitoring, and risk analysis. Then we designed a comprehensive research program including those parts of technologies and integrated it into the risk assessment system for soil and groundwater contamination.

4.1 Development of methodologies for exposure and risk assessment

The principal concept for identifying hazards and quantifying risks from contaminants in soil and groundwater environment was proposed in the first step. The major exposure pathways are direct intake from soil and subsoil, indirect intake from groundwater, indirect intake from dust and vapor in air, and bioaccumulation through food intake[8]. Almost all exposure pathways and scenarios are considered in the concept of risk assessment (Fig.3). Two types of cancer (oral and inhalation) and non-cancer risks are assumed to be the endpoints[Term3] for risk assessment. In considering exposure scenarios, health and social investigations were conducted to define Japanese default values, such as exposure factors (mean weight of person, rates of water and air intake, average rate of soil injection), and other factors of frequency and chance of exposure. In the step of exposure assessment, the rate of exposure in each pathway, and the distributions were calculated for both cancer and non-cancer endpoints. In order to define risk level, hazard information, such as the dose-response effect and the characteristic equations, was included in the system. Multi dimensional numerical models for transport analysis in subsurface media have been developed to quantify the exposure concentration.

4.2 Properties of contaminants of concern and unregulated chemicals

The accumulation of data on the fundamental properties of chemicals and the correlation between soil and pollutant is essential for completing the risk assessment. Chemical substances intended to be assessed with this system are heavy metals, volatile organic compounds, pesticides, as regulated in the soil environmental law in Japan, and other pollutants,

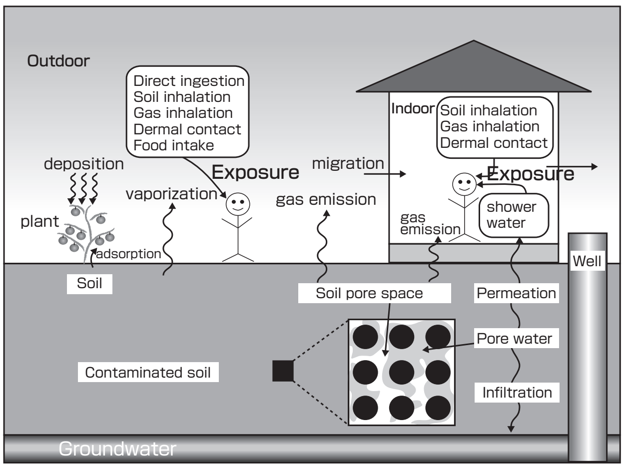

Fig.3 Exposure scenario and exposure pathways in risk assessment.

This illustrates the concept of exposure scenario in risk assessment for soil contamination. The assumption of exposure pathways, such as direct ingestion, and indirect intake from groundwater, air, and foods, is necessary for exposure assessment.

including dioxin and PCB compounds. Unregulated contaminants such as zinc, antimony, and formaldehyde, and mineral oils (petroleum hydrocarbon) are also target substances in this system. The database of the properties of 120 chemical substances and various parameters (Henry's constant, partitioning coefficients for soil-water and water-octanol) have been developed and improved through detailed examination of existing research papers and documents[9]. Because of a lack of data for petroleum hydrocarbon and PAHs (polycyclic aromatic hydrocarbons), the environmental parameters and properties, rate of vaporization, sorption coefficient, and diffusion coefficients in soil and water, have been experimentally obtained by soil column tests and partitioning examinations. Furthermore the system can be applied for complex pollutants to expand the function of the risk assessment protocol to multi components[10].

4.3 Geological information and the establishment of analytical methods

The geological information, including physical and chemical properties of soils, geological structure from topsoil to aquifer, and the behavior of groundwater flow, is very important in realistic risk assessment. In order to analyze the properties of topsoil, the existing classification method of soil types was modified using by the data obtained from geological surveys in many areas. One of the outputs in this study is a publication of geochemical and risk assessment map of subsurface soils in Miyagi Prefecture. The methods of the chemical analysis of soil components, heavy metals and other toxic substances have been established to obtain the database for both total content and leaching content[11]. Regarding the chemical and existence forms in soils and rock elution, the original method of serial extraction was developed to enable the consideration of both the toxicity and exposure rate of pollutants.

Convenient and prompt methods of soil sampling have been introduced in geological surveys at contaminated sites. The application of geophysical technique to soil contamination survey has become easier, particularly for soil sampling and contamination survey, because this technique has the advantages of low cost and low impact on the environment. As a result, geological information on the three dimensional subsurface structure and the hydrological situation has been clarified in the form of visualized data by the methods of geophysical exploration using electromagnetic and specific resistance media[12].

4.4 Analysis of soil and groundwater characteristics

In order to obtain fundamental information on the transport and migration of pollutants in soil and groundwater, some pollution surveys and monitoring were carried out for model and actual contaminated sites. The environmental parameters, such as permeability and diffusion coefficient in unsaturated soil, have been identified and analyzed. Since there was very little data on mineral oils containing various kinds of hydrocarbon, the parameters for multiphase flow and transport in soil and groundwater were measured by column experiments[13].

The chemical and biological transformations and degradations of contaminants in soil and groundwater can affect the result of risk assessment. The practical data on sorption by soil and clay minerals, chemical reactions, and biological degradation rates have been obtained on site surveys and laboratory experiments[14]. The correlation between soil and contaminants, particularly for heavy metals and VOCs (volatile organic compounds), was examined at several selected sites in Japan. In the survey of topsoil, the physical properties of soils, the properties of sorption and desorption, and the kind and content of clay minerals have been investigated to prepare databases. In the survey of groundwater, the behavior of natural attenuation owing to biological processes was investigated by long term monitoring of the contaminated soil and aquifer.

4.5 Improvement of geo-environmental information

The obtainment of geological and hydrogeological information is imperative for conducting risk assessment for specific sites, such as industrial land and urban areas. AIST developed a series of geochemistry of rocks, sediments and soils with toxic elements originated from natural sources in the general environment. Because these maps are very effective for improving the precision of risk assessment, the data for chemical components in soils and sediments have been included in the information system. The system of geochemical and risk assessment maps for subsurface soils contain numerous analysis results of the risk assessment of heavy metals, obtained by exposure distribution analysis in several particular districts[15]. The development of risk maps for heavy metals is the first trial for wide range of applications, in risk management of soil contamination, and in environmental policies related to land use. In addition, AIST and Tohoku University have developed an innovative information system for the geo-environment, named "the geo-environment informatics system", that is coupled with geological maps and other available maps of soil and groundwater under a unified code of GIS system[16].

5 Development of risk assessment system

The development of the geo-environment risk assessment system, abbreviated *GERAS*, is the final goal and output of this research, in which technologies for risk assessment have been synthesized and integrated. In developing the system, basic research on the optimum framework of scientific studies, and product realization research on model formulation, programming and numerical simulation, have been implemented by the engineering approaches of visualization and integration[17].

5.1 Integration of technology components and systematic development

The synthetic concept of the integration of technology components was firstly proposed for the completion of the risk assessment system, considering the suitable methodology, exposure model and exposure analysis for risks induced by soil and groundwater contamination. The most important motivation in the sense of systematic development is the leadership as a producer of risk assessment system. On the basis of the preliminary study on the methodologies, the types of parameters and data were selected and correlated for optimization in the form of risk assessment. In accordance with the advanced investigation, basic databases and analytic works were performed to produce a suitable risk assessment system.

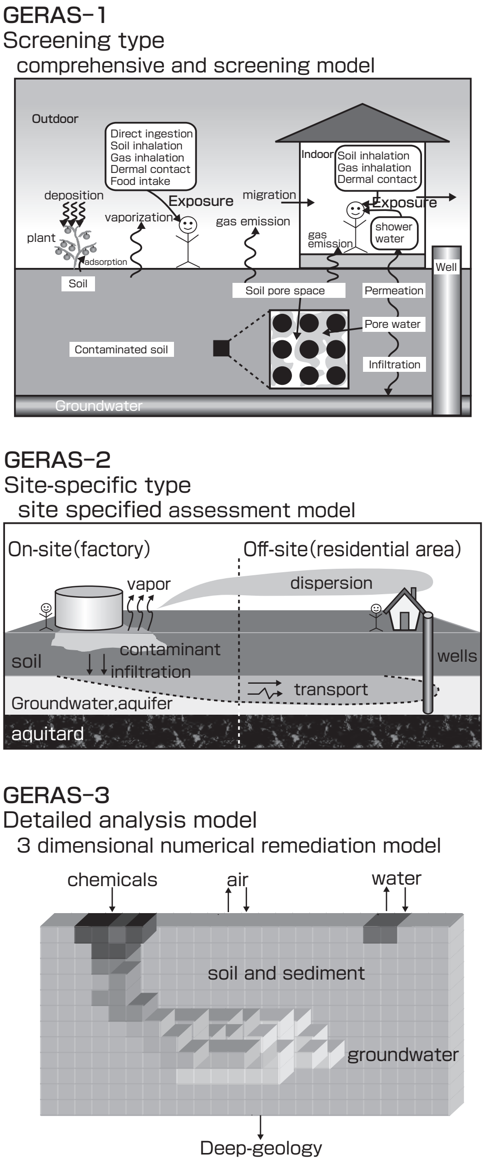

Fig.4 Layered structure of geo-environmental risk assessment system GERAS.

This illustrates three layered structures for GERAS. Depending on the risk level and purpose of risk and exposure assessment, screening type, site-specific type and detailed analytical models are installed in the system.

In the next step, the correlation and optimization of monitored and experimental data were examined to develop more reliable databases that can be utilized in the assessment. The databases have been verified using the monitoring and analytical data obtained at real contaminated sites. Default values for exposure factors and other parameters were determined and fixed for the system through modification and compensation with monitored real situations. There have been many practical cases in which monitored data and the results of risk assessment were provided in the process of feedback to the GERAS producer. Such data are very useful in improving the risk assessment system, particularly for the validation and the expansion of system functions. Several modifications of the system, such as easy input and visible output, were a result of requests from GERAS users. As shown in Fig.2, the integration of technology components and of research, characterized by a feedback process and spiral architecture, were conducted to obtain more a reliable risk assessment system.

5.2 Features of assessment system and basic functions

The features and basic functions of GERAS are illustrated in Fig.4. In consideration of the various situations of sites and purposes, the risk assessment system has three layers, screening type (GERAS-1), site-specific type (GERAS-2) and detailed analysis type (GERAS-3). On the basis of the results of basic research works, as mentioned in chapter 4, integration and coordination in technology components of risk assessment were conducted to introduce the newly designed concept in *Synthesiology*. Typical assessment cases for heavy metals and VOCs are shown to explain the features of components and technical breakthrough in developing the GERAS.

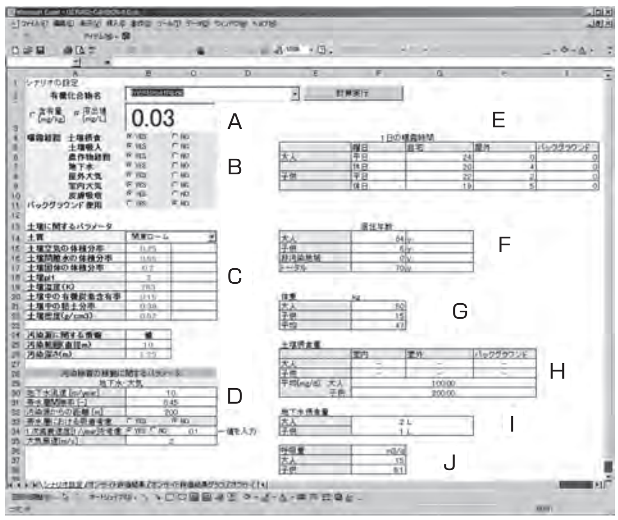

Fig.5 Configuration of input data and parameters in the risk assessment system.

This shows the configuration of computer system and input parameters needed for risk assessment system, GERAS-1,2. The properties of soil and groundwater, parameters for transport of pollutants, and exposure pathways and exposure factors are necessary.

GERAS is a system that operates on a Windows computer system, as described in Fig.5. This system has various functions of exposure and risk analysis, such as the correspondence with the types of soils inherent in Japan, exposure factors typical to the Japanese, and user input parameters such as soil leaching concentration and organic content in soil samples. In the actual operation of the system, user first inputs the kind of chemical substance as the target pollutant in soil (Fig.5, A), selects exposure pathways (Fig.5 B) that are possible to expose through them, and sets basic parameters. Then the user fills out some columns; soil parameters obtained from site-specific survey (Fig.5 C), groundwater parameters obtained from monitoring data (Fig.5 D), and other parameters for the receptor (Fig.5 E-G). The system has a default dataset of soil parameters and exposure factors, soil ingestion rate, water intake rate and inhalation rate for the average Japanese (Fig.5 H-I). The major exposure pathways considered in GERAS are direct intake from soil ingestion and indirect intake from drinking water, air and foods. The paths of inhalation from vaporized and particulate matters from contaminated soil, and dermal contact with contaminated soil and groundwater are also targets of risk assessment.

The calculation of exposure and risk analysis begins after the completion of data input. This system calculates the partitioning behavior and distributions of specified chemicals in soil, pore water, and vapor phases. By taking the initial condition of soil leaching value in contaminated sites, the concentration in soil vapor, and pore water can be estimated using pH, total organic content, and sorption factors of specified soil. In the next step, the exposure concentrations in soil, water and air are determined by transport analysis in every environmental medium, such as air, groundwater, crops and others. The rate of human exposure in each pathway is calculated for an exposure scenario in consideration of exposure factors and the properties of receptor. Finally, risks for endpoints are summed for all exposure pathways, on the basis of the toxicity data of chemicals. The calculation of risk is prescript with two categories; cancer risk and noncancer risk. The probability of cancer (10^{-6} or 10^{-5}) is assumed to be the criterion for chemicals related to cancer risk, and the rate of exposure compared with tolerable daily intake (TDI) is one of the criteria for noncancer risk.

5.3 Cases of risk assessment by GERAS

Practical risk assessments were carried out using GERAS, as described below for a heavy metal (arsenic compounds: As) and VOCs (trichloroethylene: TCE) [18]. These chemical substances are toxic to humans; As has the possibility of being carcinogenic, and TCE is the cause of hepatopathy.

In the risk assessment of these cases, the concentrations of the substances in contaminated soils were assumed to the standard values that are regulated in the soil contamination countermeasures act in Japan; total content of 150 mg/kg for As, and leaching value of 0.03 mg/l for TCE. The soil properties were obtained by the investigation of Kanto loam. The following exposure scenarios were assigned, type of land use: housing site, duration of exposure: 6 years for children and 64 years for adults, frequency of exposure: 24 hours per day. The exposure pathways were considered to be realistic worst cases; direct ingestion of soil, indirect inhalation (indoor and outdoor), dermal contact, groundwater intake, and ingestion of crops and foods grown in affected soil. The average exposure through a lifetime of 70 years was calculated for each chemical substance and pathway. World Health Organization (WHO) has defined the standard or tolerable amount of exposure for each chemical. According to the WHO reports, the tolerable daily intake (TDI) is 2.1 μg/kg/day for As, and 24 μg/kg/day for TCE. Taking the possibilities of exposure from the atmosphere and hydrosphere into account, 10 percents of the TDI was regarded to be the target value of the exposure baseline in this assessment; 0.21 μg/kg/day for As, and 2.4 μg/kg/day for TCE. The monitored and analyzed data were used to clarify the characteristics of soil and groundwater.

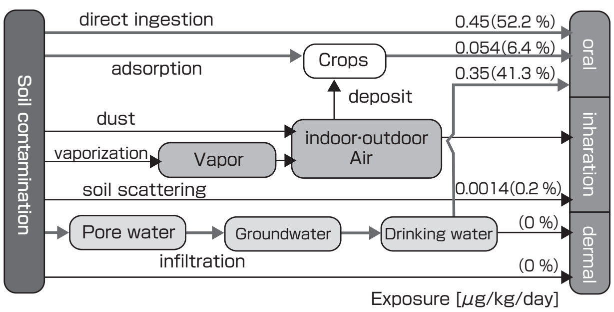

Fig.6 The result of exposure rate and partition from contaminated soil by arsenic.

As s result of exposure analysis using GERAS-1, this represents the rates of exposure and the partition for each pathway from As contaminated soil. The major pathways in case of arsenic are direct ingestion and groundwater intake.

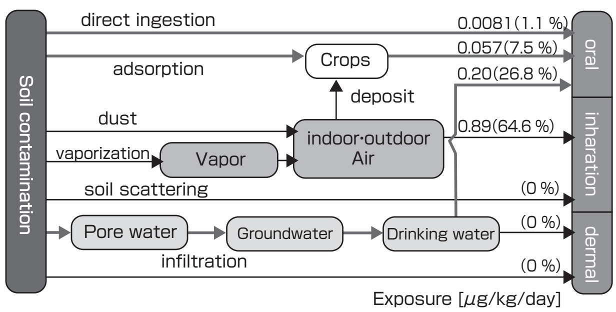

Fig.7 The result of exposure rate and partition from contaminated soil by TCE.

As s result of exposure analysis using GERAS-1, this represents the rates of exposure and the partition for each pathway from TCE contaminated soil. The major pathways in case of TCE are inhalation from indoor and outdoor air (atmosphere) and groundwater intake.

Fig. 6 and Fig.7 show the results of exposure assessment for As and TCE, respectively. In the case of As, the major pathways of exposure are direct ingestion and groundwater intake. However, inhalation from air indoors and outdoors is dominant for TCE. These results of exposure assessment show that the average exposure rate cumulated for all pathways is 0.85 μg/kg/day for As and 0.76 μg/kg/day for TCE. Compared with the target value of the exposure baseline, the exposure rate of As is about 4 times the exposure baseline, indicating that the risk level for this situation exceeds the baseline, which is the criterion in terms of human health. On the contrary, the risk level of TCE in this situation is considered to be acceptable for maintaining human health, because of much lower exposure rate compared with the baseline.

Although the rate of exposure and the risk level can be analyzed using GERAS, this system can also provide the target concentration for the remediation of soil and groundwater by inverse analysis. The case study on As-contaminated soil showed that the risk level was higher than the criterion. By the inverse analysis of that case, the target total content of As in soil can be estimated to be 37 mg/kg. In order to achieve this target, we should adopt measures of remediation or some form of risk management. The control of major exposure pathways is an effective measure in terms of risk mitigation. In the case of As, direct ingestion and groundwater intake are principal pathways, so that risks can be reduced by interrupting these exposure pathways, by adopting anti-scattering measure for soil and/or switching to a safe source of drinking water.

5.4 Disclosure and widespread utilization of developed assessment system

The development of the screening type model (GERAS-1) and site-specific type model (GERAS-2) was completed after a review by experts, and was disclosed to the public in February 2006. A CD-ROM with the software of the risk assessment system and databases has been distributed to users wishing to utilize the system, along with a user manual and confidential serial number. More than 800 units have been distributed and utilized by various users and industries, as illustrated in Fig.8, business sites, factories, companies for remediation and analysis, geological consultants, universities, and local governments. Major intended purposes are the self-governance of soil and groundwater contamination in industrial land and factories. The English version of GERAS is available for overseas use, particularly to Asian countries (China, Korea, Thailand and Vietnam), Europe and USA[19]. A special version of GERAS for mineral oils (gasoline, kerosene, and petroleum fuel) will be made available in 2009. Another system of detailed analysis, GERAS-3, for risk assessment and risk mitigation during the transport and transformation of environmental pollutants is under development, and will soon completed as a part of the whole GERAS.

The functions of GERAS have been improved and the databases have been renewed and modified using feedback data from users who applied GERAS to actual contaminated sites. Such risk assessment results are highly valuable in amending the system because the properties of soil and groundwater exhibit great variation and the results of risk assessment have great differences in time and space. The applicability of the developed risk assessment system is validated through the accumulation and amendment of data and the results of site assessments, and the system has been continually modified through version upgrades. So far, the modification of soil and groundwater databases, the addition of newly regulated chemicals (fluoride and boron), the application to unregulated pollutants (mineral oils, biofuels, MTBE, ETBE and PAHs), and the use the leaching value, have been implemented in several version upgrades in the continuing development of GERAS.

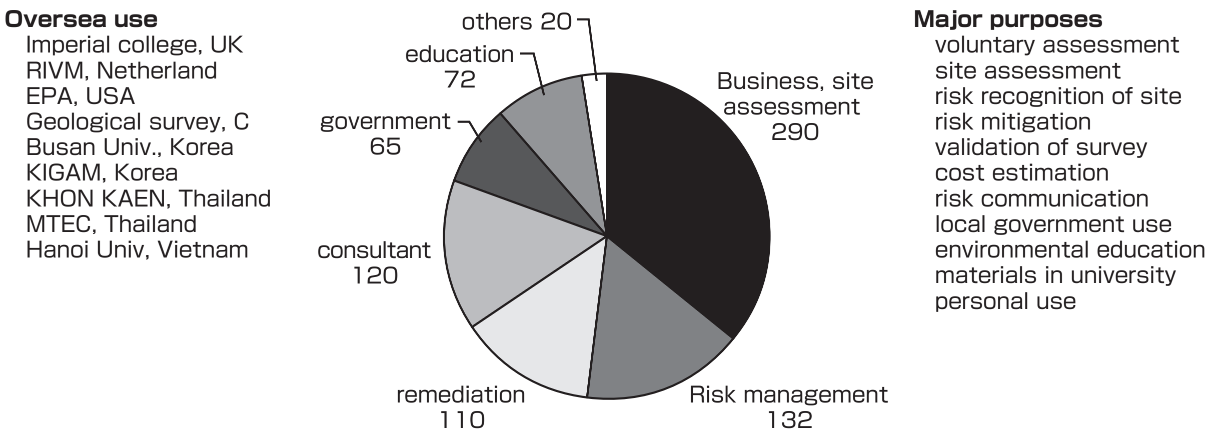

Fig.8 Category of business sectors and organizations where GERAS is distributed and utilized, and major purposes of application.

This diagram shows the number of business sectors and organizations where GERAS have been utilized. Among the whole of about 800 units, the most frequent users are business places and factories. It has been widely utilized for the purpose of remediation action and geological consulting.

6 Further technical tasks and application to society

The application of the development system to various types of environmental problems and the process of its establishment as a social system, including the contributions to industry and society, are described in this chapter.

6.1 Further technical tasks

As described in chapter 5, the developed GERAS comprises various technology components and databases. The optimum configuration of these parts and the reliability of databases have been verified in the previous research, but further improvement will be necessary, particularly for upgrading the risk assessment technology. For this purpose, further research will be continued for the advancement of basic studies on uncertainty of risk assessment and the advanced statistical computing method. These studies are essential for the public acceptance of risk assessment technology for environmental problems, which must be implemented to achieve more transparent risk communication. In order to advance social acceptance, the improvement of the reliability of the assessment results and databases is of grea+t importance, for example, by increasing cases of application at actual contaminated sites. The principal process of this research development involves a loop-type structure of improvement, as illustrated in Fig.9, in which fundamental databases of input side should be amended using the assessment result output. A couple of loops represent a scenario of both risk assessment and environmental improvement, including risk reduction, control, and management in each process. The accumulation and verification of data obtained through appropriate geological survey and analysis are most important to maintain the quality of risk assessment, as shown in the lower part of Fig.9. Through the introduction of these multilayered spiral structures in risk assessment, the benefits of both risk mitigation and cost reduction can be obtained.

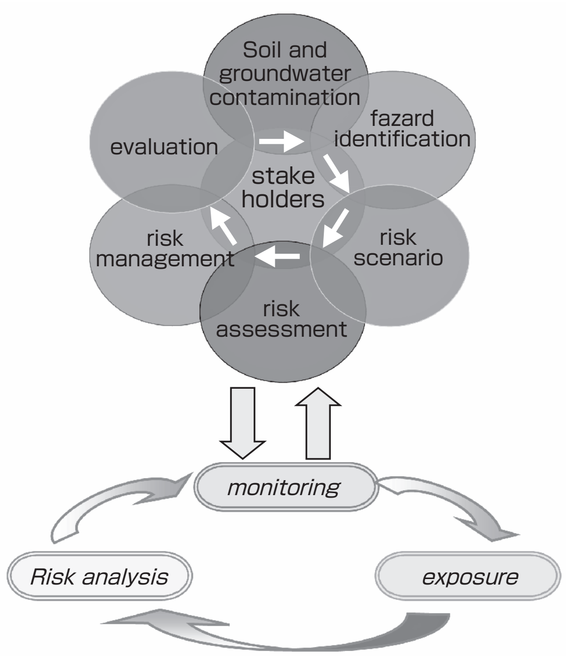

Fig.9 Looped scenario in risk management and multilayered spiral structures.

This illustrates a discriminative looped scenario in risk management for soil and groundwater contamination and a spiral process. In the implementation using GERAS, these multilayered structures are very important for risk mitigation.

GERAS has the possibility to be expanded to more generic environmental problems, such as ecological risk assessment and global environmental issues. The preliminary investigations on the application of GERAS to aquatic life and microorganisms have already been started, in order to assess risks for small amount of toxic elements. Most of the technology components can be utilized in the same manner as for geo-environmental problems. In addition, the risk assessments in the cases of geological CO_2 capture and storage and nuclear waste storage underground might be similar to the developed methodology of risk assessment. The trials of concrete investigation have been begun to clarify the above possibility.

6.2 Contribution to countermeasures for soil and groundwater contamination

The greatest benefit obtained by the application of the developed system is voluntary environmental improvement with a scientific understanding of risk assessment. The comparison of risk levels before and after the remediation provides important information for risk management and risk communication, on the basis of the scientific risk analysis for specified contaminated sites and various situations. Because GERAS can provide information on the exposure rate and risk for each pathway, it will be possible to control the exposure and to mitigate risk in any situation. The implementation of risk assessment contributes to a large cost reduction in risk management, for example, through the selection of an appropriate remediation method and the shutoff of the exposure pathway, compared with the conventional regulation of uniform environmental criteria.

Fig.10 represents the estimation results of risk mitigation and cost reduction upon the introduction of the risk assessment methodology. The reduction of environmental risk and cost of countermeasures is estimated to be 50 percent in the present situation, since the method of risk-based management has already been introduced in industrial sectors[20]. The application of the developed risk assessment technology can largely contribute to risk management, and ultimately reduce the cost as a result of its introduction into social and legal systems. The mitigation of environmental risk can also be expected by the exposure control and the development of low-cost remediation technologies such as an advanced method using geo-microbiology. In practical environmental management at industrial sites, GERAS has been widely used, particularly for voluntary risk management. Thus the risk assessment technology enables the realization of both environmental improvement and cost-effective countermeasures for soil contamination. Therefore, the establishment of a paradigm and the scientific methodology contribute to society and industry in terms of the economical aspect concerning cost benefit.

On the other hand, this system can contribute to alleviating the social issue of risk communication, transparent information recognition and reasonable decision-making. To clarify the real situations, the exposure and risk under specified conditions, the risks between before and after remediation, and the cost effectiveness as the relationship between cost and risk, more reasonable and scientific risk management can be expected. The smooth conversion of land use would be possible from unused industrial sites, named “brown fields”, as the basis of guidelines formulated from risk assessment.

6.3 Overview of the establishment of social system

In order to realize the establishment of the risk assessment technology in the social system, there are several technical and social tasks to be completed in future work. One of them is the technical methodology to ensure transparency and reliability of the risk assessment system. In the development of the system, efforts to realize the smooth application to industry and society have been taken by interactive communication between the producers and users. The introduction of this system into the environmental management system (EMS) is a favorable way to promote its widespread utilization. In the procedure of EMS, the methodology of site assessment is given for voluntary risk management at industrial sites. Since there is no prescription on how to conduct risk assessment in Japan, the developed GERAS has been widely used for the purposes of contamination assessment and evaluation of remediation action[21]. One of the important outcomes is the introduction of GERAS into the social recognition system of “Environmental Site Assessment”, operated by the Japan Environmental Management Association for Industry (JEMAI). The risk assessment technology and GERAS have been accepted for the risk assessment system of JEMAI, in order to promote its utilization of environmental business in industry[22].

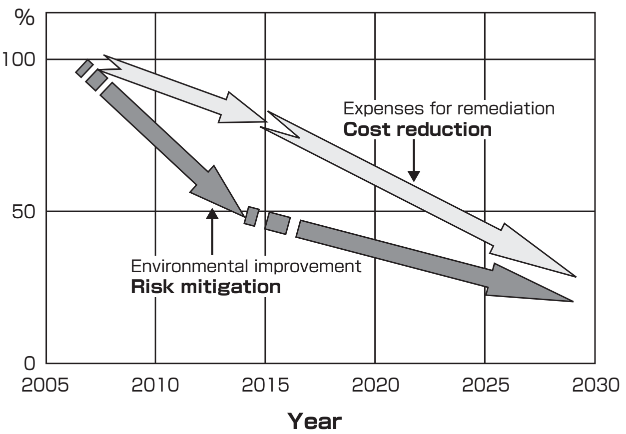

Fig.10 Estimation of risk mitigation and cost reduction caused by the practice of risk management.

This shows the estimated results and trends on the reduction of environmental risk and remediation cost, after the introduction of risk assessment technology. It is expected to mitigate risk and to reduce cost by more than 50 percent to the year of 2025[20].

The investigation for the amendment of the soil contamination countermeasures act in Japan is under review by authorities. According to the official report, “Overview of the future soil environmental management by advisory body”, presented by the Ministry of the Environment of Japan in March 2008[23], the necessity of a reasonable and appropriate risk management regarding site-specific land utilization was stated to promote cooperation between the legal system and voluntary system in industry. The fundamentals in promoting the law amendment are risk assessment technology and the paradigm of risk governance, obtained in our research work. The importance of site- specific risk assessment and reasonable risk management has been accepted socially, and the methodologies will spread into the social system.

In April 2008 we demonstrated GERAS at the world industrial exhibition of “Hannover Messe”, where many researchers and industrial managers in Europe expressed interests in the application. Compared with the U.S. product[24], GERAS has some obvious advantages; the development as a user-specific system, the possibility of data feedback from users, and the abundance of databases for soil and groundwater[25]. Therefore, there were many responses from foreign countries wishing to utilize the system, as listed in Fig.8. As tasks in the near future, new functions of risk assessment will be installed in the continuing improvement of GERAS, to promote research output toward not only domestic but also the worldwide users.

7 Conclusions

The risk assessment technology for soil and groundwater contamination was established by the integration of technology components from various research areas. The risk assessment system, GERAS, was developed in this research program, on the basis of a unified methodology of risk assessment and databases. In this research, we introduced an advanced methodology of risk-based assessment and the paradigm of risk governance, in order to combine and integrate optimum technologies under the concept of a spiral process and architecture. As a result, the development of the first risk assessment system for soil and groundwater contamination in Japan was completed, and the product was widely distributed and utilized for use in voluntary management by industry and local governments. This system will soon be introduced into the social system, enabling the implementation of a reasonable risk management, and will contribute to the sustainable development of society and industry. The widespread utilization of the developed system will be promoted, since there are many advantages of this system compared with products from foreign countries. Moreover, the research work will proceed in order to develop a geo-information system for soil, groundwater and geology, as a public property needed for the operation of the risk assessment system.

Terminology

Term1. *Type 2 Basis Research*: This research is a research type where various known and new knowledge is combined and integrated in order to achieve the specific goal that has social value. It also includes research activities that develop common theories or principles in technology integration (see page i).

Term2. *Product Realization Research*: This is a research type where the results and knowledge from *Type 1 Basic Research* and *Type 2 Basic Research* are applied to embody use of a new technology in the society (see page i).

Term3. Endpoint: This is the target condition or situation in the assessment exposure and risk. In the case of human risk, it is a concrete event, such as the occurrence of lung cancer caused by oral inhalation or kidney damage due to oral intake of toxic substances.

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Discussions with reviewers

1 Significance in *Synthesiology* of this research as *Type 2 Basic Research* and *Product Realization Research*

Question & comment (Akira Ono)

I think this research is an excellent work intended to solve the problems of complex correlation between risk and environment by *Synthesiology* approach of *Type 2 Basic Research*. The completion of the risk assessment system, which enables its utilization in society, has great value in terms of *Product Realization Research*.

Question & comment (Shigeko Togashi)

This research presents a scenario for a research strategy to be regarded for an outcome obtained by a *Synthesiology* process. This paper is favorably evaluated, because it provides a good example of its application to industry in a study of *Synthsiology*, and presents future tasks and prospecting ideas.

2 The correlation among toxicity, exposure and risk

Question & comment (Akira Ono)

In the paper, there are the technical terms of exposure, risk and toxicity. What is the relation among the terms and their meanings?

What is the real situation of direct ingestion and inhalation as exposure pathways, where such intake or ingestion may occur in the situation of direct intake from contaminated soils, and how much exposure has been considered in the cases of ingestion and inhalation? Is the exposure rate negligible or not in terms of human risk?

Answer (Takeshi Komai)

The meanings of the terms are explained in figure 3. Exposure is defined as the rate of intake of chemical substances through environmental media. Risk is the result of exposure, as defined in the form of the probability of adverse effects to health and/or the environment. Risk can be calculated by the product of toxicity and the rate of exposure, or by other regulated methods in the case of cancer risk assessment.

Direct ingestion of soils is the situation that a human intakes soil grains during field work and/or when playing with soil and sand. The exposure due to direct ingestion is considered to be a large amount in the case of playing outdoors in childhood. According to the formal data in a paper of dioxin risk assessment reported by the Ministry of the Environment, the average intake of soil is assumed to be 100 mg/day and 200 mg/day for an adult and child, respectively. The exposure due to inhalation is the intake of soil particles in a common environment and during works in a garden. The estimation of exposure and risk for heavy metals (Pb, lead) shows that the rate of direct ingestion is more than 50 percent and that of inhalation is about 5 percent. The rates in these pathways are quite large relative to the total amount of exposure from contaminated soil.

3 Introduction of a common criterion or unified index

Question & comment (Akira Ono)

In chapter 3, the significance of a common criterion or unified index is discussed. In conducting the *Type 2 Basic Research* where several technological elements are selected and synthesized into a form of product, the incorporation of such a common criterion and unified index is one of the important points.

What are the concrete objects of the common criterion or unified index in this research? Please explain also the background and concept behind the introduction of these terms.

Answer (Takeshi Komai)

The existing criterion is a uniform standard or target value for maintaining human health in the environment. However, the situation of very little risk to humans should be possible when the land is well managed and monitored for contaminated soil and groundwater. Thus we introduce the risk-based assessment for quantifying a specified situation. The term "common criterion" defined in this research is "exposure obtained by risk assessment". The rate of exposure from contaminated soil differs very greatly depending on the land utilization, properties of soil and groundwater, and lifestyle. The term "unified index" is defined as "human risk of exposure to chemicals from the environment". The methodology to assess the exposure precisely is a general and scientific method used to make decisions on the effects on humans and the environment.

4 The reliability of the result of risk assessment

Question & comment (Akira Ono)

As mentioned in chapter 6.1, the reliability of results obtained from the developed risk assessment system is of great interest to users, particularly the methodologies and databases utilized in this system. Since the reliability of results depends on the uncertainty of risk assessment, what do you think about the major sources behind the uncertainty of the risk assessment result? Please explain the possible sources for both methodology and databases.

How much uncertainty, roughly what percentage, do you estimate for the final result of risk assessment for As and TCE, as shown in section 5.3?

Answer (Takeshi Komai)

In order to ensure the reliability of the risk assessment result, it is essential to improve the precision of applied exposure models and the quality/quantity of databases in risk assessment system. The developed risk assessment system has already been well evaluated by experts on the verification of the reliability of assessment results. In addition, the system has been validated using feedback data and the results of risk assessment at actual sites. The quality of risk assessment depends on two factors; the uncertainty due to the lack of information or data, and the variability relevant to the inherent diversity. In this assessment, the major sources behind the uncertainty are based on the properties of soil and groundwater. It should be possible to reduce the uncertainty if we can carry out a sufficient number of geological and geophysical surveys. Since the variability of factors is not considered in this assessment, the level of uncertainty can be estimated to be less than 5 percent in the final results of As and TCE risk assessment.

5 The extension of the developed risk assessment system to ecosystem issues

Question & comment (Akira Ono)

Expansion of the risk assessment system developed for soil and groundwater environment to other applications may be possible. What are the future tasks in solving the problems, if we intend to expand the system to the risk assessment for the ecological system?

Answer (Takeshi Komai)

The methodology for exposure and risk assessment has general-purpose features that enable its extent to other targets, such as the atmosphere, hydrosphere and global environment, such as an ecosystem. We already intend to expand the developed system to a broad range of utilizations, such as the assessment of the environmental impacts of chemicals on aquatic and plant life. For such a development, it is necessary to overcome the problems of clarifying the intake mechanisms for plants and microbes and the endpoints for assessing the diversity in ecosystem. Since the linkage with other exposure models for the atmosphere, rivers and the ocean is necessary, we will conduct collaborative research and development with research groups in AIST and others.

6 The features in *Synthesiology* research and relevant research areas

Question & comment (Shigeko Togashi)

Please clarify the relationship between the table and figure 2, which is a good explanation of the trial of *Synthesiology* research.

Answer (Takeshi Komai)

In order to explain the features and importance in *Synthesiology* research, the process of integration among research areas and their contributions are newly described in figure 2. The list of research areas, technology components is amended in the table to clarify the relationship between the purposes and academic fields.

7 The spiral structure in risk management and risk assessment

Question & comment (Shigeko Togashi)

Some erroneous uses of the technical terms risk management and risk assessment may exist in the text, where both terms have vague definitions and relations.

Answer (Takeshi Komai)

Some misuses of the technical terms risk management and risk assessment were corrected to their appropriate uses in the text of the paper. The description of complicated structures in figure 9 was amended to simplify the relationship between risk management and risk assessment.

The loop-like structure is described in figure 9 to illustrate the process of risk management. In order to understand the whole spiral structure of risk management, the figure was simplified and modified into multiple spiral structures.

Development and application of photocatalytic technology

— Industrialization of sustainable eco-technology —

Hiroshi Taoda

[Translation from *Synthesiology*, Vol.1, No.4, p.287-295 (2008)]

Photocatalysts are capable of decomposing organic compounds such as toxic and bioresistant pollutants into non toxic gases in the presence of light. Material development and application of a number of photocatalysts have been carried out taking into consideration many factors including application, cost, as well as applicable regulations. As a result of these efforts, a variety of new photocatalytic products are now on the market.

Keywords : Environmental purification technology, photocatalytic technology, titanium dioxide (TiO_2), water purification, air purification, antibacteria and antifungi, antifouling

1 Background of photocatalytic technology development

Recently, environmental pollution by hazardous chemical substances including dioxin from incinerators, organotin compounds used in ship bottom paint, PCBs, agrichemicals, solvents, and detergents is occurring at global scale, and it has become serious issue that threatens human survival. These chemicals include substances that are harmful at extremely low concentration of ppb level. They pollute water, air, and soil at global scale, and are very difficult to remove.

Conventionally, treatment of environmental pollutants was done with pyrolytic method that involved collection, concentration, and incineration. However, purification of the environment using this method necessitates treatment of vast quantity of water, air, and soil since the pollution is spread widely. To accomplish such treatment, large amount of energy such as fossil fuel is necessary, and use of such energy produces large amount of carbon dioxide. Also incineration process increases the danger of production harmful substances such as dioxin. Therefore, attempts of removal of environmental pollutants spread widely at low concentration throughout the environment using conventional technology may promote energy crisis, global warming, and further environmental pollution.

The technologies for measurement and analysis of environmental pollution have progressed and the state of environmental pollution is becoming clarified, but development of technology to repair and purify pollution at global scale progressed slowly. Sustainable environmental purification technology, where the environment can be purified without use of fossil fuel or harmful chemicals, was eagerly awaited.

The development of sustainable environmental purification technology is obligation of advanced nations that had been emitting large amount of environmental pollutants. It is an industrial technology that must be developed by Japan that claims to be the leading nation of science and technology. Development of the technology, commercialization, and diffusion of the products will contribute to future development of Japanese industry, and will improve global environment.

Photocatalyst can decompose and detoxify harmful organic chemical substance into carbon dioxide and water upon irradiation of light. Using the photocatalytic technology developed, the environment can be purified without using fossil fuel or harmful chemicals. Wide-ranging application is possible to water treatment, deodorization, VOC treatment, air purification, antifouling, antifogging, antibacteria/fungi, freshness maintenance, and dioxin removal.

While photocatalyst can be applied to various environmental pollutions and is effective method for global environment purification, many problems needed to be solved including technical issues such slow reaction speed, difficulty of handling, and difficulty of application to textiles and plastics, as well as issues of economy and reliability.

2 Objectives and goals of photocatalytic technology development

Photocatalyst is a substance that assumes high-energy state by absorbing light and causes chemical reaction by supplying that energy to reactants. Semiconductors and metallic complexes are used as photocatalysts, and most frequently used substance is titanium dioxide (TiO_2). Titanium dioxide is widely used as pigment. It is safe and nontoxic with

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excellent durability, and is used in toothpaste and cosmetics, and is approved as food additive.

When titanium dioxide is irradiated with light, electrons with negative charge and holes with positive charge are formed, just like in silicon used in solar cells, as shown in Fig. 1. These electrons and holes have extremely powerful reduction and oxidation actions, and active oxygen such as OH radical and super oxide anion (O_2^-) are formed by reaction of water and dissolved oxygen. The holes and OH radicals have particularly strong oxidative power. While the bonding energies of carbon-carbon, carbon-hydrogen, carbon-nitrogen, carbon-oxygen, oxygen-hydrogen, and nitrogen-hydrogen bonds in the molecules composing the organic substance are 83 kcal/mol, 99 kcal/mol, 73 kcal/mol, 84 kcal/mol, 111 kcal/mol, and 93 kcal/mol, respectively, the energies of holes and OH radicals are significantly higher at 120 kcal/mol or above, so it can easily break these bonds.

Due to this action, various harmful organic substances including toxic chemicals dissolved in water and chemicals in air such as malodorous substances can be easily decomposed and detoxified by irradiating with light. Moreover, there are several advantages such as ability to treat dispersed environmental pollutants safely, efficiently, and semi-permanently without using harmful chemicals or fossil fuels, instead using clean and inexhaustible sunlight.

In this research, low-cost, high-performance photocatalyst was developed for actual use and specific purpose such as water treatment, air purification, antibacteria/fungi, and antifouling. The developed photocatalyst was used in practical application to various environmental fields, and the goal is to purify the earth environment through diffusion of photocatalytic products.

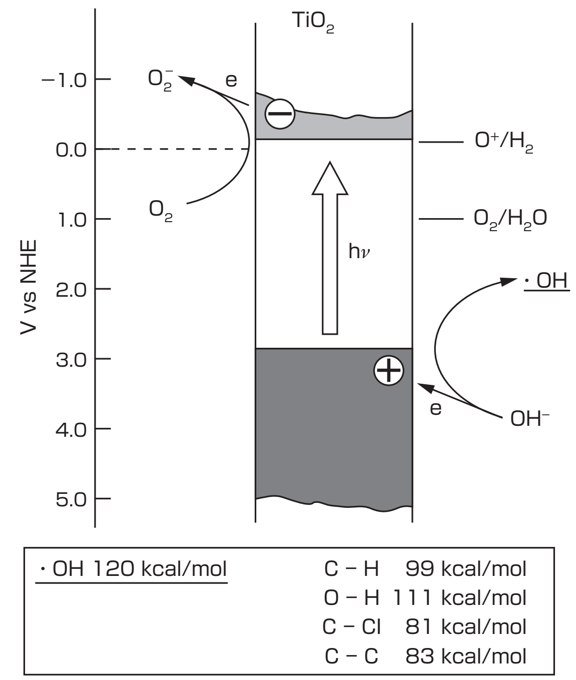

Fig. 1 Energy level and photocatalysis of titanium dioxide.
V = electrical potential, NHE = normal hydrogen electrode. V vs. NHE is potential against normal hydrogen electrode, or potential when normal hydrogen electrode is used as standard. hν = energy of light.

3 Research scenario toward accomplishment of goal

It is said that there is "valley of death" that must be overcome to achieve commercialization of new technology. Until now, only the technological aspects or technological breakthroughs were mentioned in the valley of death, but there are also economic and social aspects.

To accomplish realization, there exist not only technological valley of death such as improvement of performance, but economic valley of death such as cost performance against existing technology for same purpose, and social valley of death such as maintaining reliability and safety that are necessary for use and acceptance by consumers. Therefore, to actually apply and realize photocatalytic technology toward commercialization, it was necessary to overcome the three valleys of death including technological, economic, and social valleys.

3.1 Technological valley of death

Photocatalysis was known before the World War II as phenomenon of paint degradation where white pigment containing TiO_2 flaked off when it was exposed to sunlight, and this was considered a problem for long time. Therefore, pigment manufacturers that manufactured titanium dioxide spent effort to prevent degradation by coating the surface of TiO_2 pigment with ceramics that do not undergo photocatalysis.

In the 1950s, Fujio Mashio, Shinichi Kato, *et al* of the Kyoto Institute of Technology engaged in research to utilize photocatalysis reaction in purification of environment and synthesis of useful substances[1]-[3]. This was followed by researches around the world on decomposition and detoxification of harmful organic chemical substances using photocatalyst. Experiments were done with various environmental pollutants such as hydrocarbon, organic chloride compounds, agrichemicals, and synthetics detergents, and there were many reports on successful decomposition and detoxification.

However, fine particles were used in these experiments, and practical application did not progress due to disadvantages such as difficulty of separating treated water and photocatalyst, and only batch treatment could be done since the photocatalyst dispersed in water in water treatment. To advance practical use of photocatalyst, it was mandatory to fix the catalyst onto a substrate.

Photocatalyst also had limitation that it could not decompose the target substance unless it came close to the surface, and there were problems of slow reaction speed that posed difficulties in removal of pollutants in high concentration and short treatment time. In addition, when the catalyst was attached to textiles or plastics, the materials were broken down by photocatalysis, and therefore use in textiles and plastics was impossible. Although photocatalyst could be used widely in environmental field, different forms needed to be devised according to use (for example, surface should be smooth for antifouling, while surface should be uneven to increase surface area for deodorization), and it was necessary to develop photocatalysts that matched specific use for practical application and product realization.

Development of effective photocatalyst and photocatalytic products adapted to actual use was the technological valley of death.

3.2 Economic valley of death

Although photocatalytic technology can be widely applicable as mentioned above, cost performance becomes an issue when compared with existing technology.

Titanium ranks 9th in abundance in the earth crust. Titanium dioxide is used as pigment, and it is abundant and low cost as resource. However, titanium dioxide used in photocatalyst is generally nano-sized, ultra-fine particle and is ten times expensive compared to rougher TiO_2 particles used in pigments. Also, it is necessary to use artificial light source for nighttime use or device fabrication. To put photocatalytic technology, which is a novel technology, to practical use, it is necessary to lower the cost to less than or close to the cost of existing technology, or to provide advantage over existing technology. To promote wide use of photocatalytic technology, it must be usable in large scale at civil engineering sites.

Therefore, development of photocatalyst and photocatalytic products with cost performance that allows replacement of existing technology, and development of photocatalyst and photocatalytic products that can be mass-supplied at low cost were economic valley of death.

3.3 Social valley of death

No matter how excellent the technology, it is useless unless it is used in the society. Products that use photocatalysts have characteristic that the effect cannot be perceived immediately. For example, self-cleaning effect takes several months after installation before the effect becomes visible. Therefore, many fake products were marketed and proper products failed to win confidence of the consumers. Also, due to lack of uniform and reliable testing method to evaluate the performance of photocatalysts, comparisons could not be made, and this was a barrier to development of high-performance photocatalyst. Therefore, education activities for photocatalytic technology and standardization of performance evaluation tests were necessary. Since photocatalysts differ in performance depending on use, it was necessary to develop performance tests for different use. The evaluation test was mandatory as “scale” in developing high-performance catalyst.

Photocatalysts must gain confidence of the consumers before they could be accepted in the society. It was also necessary to follow safety and environmental regulations such as RoHS (Restriction of Hazardous Substances) Directive and the WEEE (Waste Electrical and Electronic Equipment) Directive of the European Council for hazardous substance in electric and electronic appliances, as well as Pharmaceutical Affairs Law, pollution laws, and PRTR (Pollutant Release and Transfer Register). This was the social valley of death.

3.4 Scenario to achieve the goal

The aforementioned three valleys of death had to be overcome before photocatalytic technology could be accepted in the society. The following scenario was considered to overcome them.

First, for technological valley of death, technological development is generally considered to take a linear course from *Type 1* to *Type 2 Basic Research*, and then to development and practical application. However, in photocatalytic technology, there was almost no result of *Type 1 Basic Research* 25 years ago when we started research.

Although Honda-Fujishima effect is extremely famous as principle of photocatalysis, this was actually hydrolysis by photoelectrode reaction of titanium dioxide and did not involve the use of photocatalyst. Also, not much was known about the photocatalysis mechanism except that electrons and holes were produced by irradiation to cause photocatalysis reaction. The only method known to increase performance and reaction speed of photocatalyst was to prevent disappearance of electrons and holes by rebonding, and nothing much was known on how this could be done.

Therefore, high-performance catalysts for particular usages were developed by trial and error by imagining the actual use scene. The substances that must be added to create high-performance catalysis from TiO_2 base were sought by trial and error. Then we developed specific-purpose high-performance photocatalyst by finding the additive, conducted use application, developed higher performance photocatalyst by feeding back the result, and progressed to new application using the higher performance photocatalyst.

In this case, R&D of photocatalyst was interdisciplinary, and it was necessary to collaborate with technologists and companies that possessed advanced technology as well as specialists of several fields. We encouraged their participation, and conducted R&D strategically and collaboratively.

To overcome the economic valley of death, we developed low-cost photocatalyst and photocatalytic product based on TiO_2 that were inexpensive, safe, and mass-suppliable, using industrial waste as matrix. We worked on application and development with advantage unseen in existing technology. This enabled production of environment purification material from industrial waste, and contributed to recycling industrial waste. It also enabled unique applications untried before.

For social valley of death, we organized research groups and industrial organizations for photocatalyst to standardize evaluation test that was necessary for development of high-performance photocatalysts. We also worked on promoting photocatalytic technology to society through organization of and participation to exhibitions and seminars. We developed photocatalytic technology considering regulations for safety, environment such as RoHS and WEEE, Pharmaceutical Affairs Law, pollution laws, and PRTR laws.

Through these strategies, we attempted to overcome the three valleys of death including technological, economic, and social valleys.

4 Execution of R&D toward practical application of photocatalyst

4.1 Development and application of high-performance photocatalyst

I shall describe the actual R&D and application in chronological order.

4.1.1 Development of TiO_2 transparent film photocatalyst [4]

Photocatalyst has limitation that decomposition does not occur unless the target substance comes in contact upon irradiation, and efficiency increases with larger surface area since photocatalysis occurs on the surface. Therefore, high activity titanium photocatalyst in form of ultra fine powder with small particle diameter yet with high surface area had been developed. However, photocatalyst in particle form has disadvantage of being difficult to handle and to collect as they are blown away by wind. It was mandatory to fix the TiO_2 photocatalyst onto substrate for practical use, and various methods were attempted.

In the method of fixing by mixing TiO_2 powder with organic binders, there was problem with durability since organic material that was used as adhesive was decomposed by photocatalysis and TiO_2 powder gradually dropped out. In method where TiO_2 powder was mixed with inorganic binders such as cement or glaze, the TiO_2 became embedded and light could not reach. The target chemical substance could not come in contact with TiO_2 and the photocatalytic performance decreased. Other methods included CVD, PVD, and sputtering, but they had problems where large surface area was difficult to manufacture since vacuum container was necessary, and the process required large amount of energy.

Therefore, we developed photocatalyst in film form consisting only of TiO_2 fixed onto substrate using sol-gel method that can be conducted simply at low cost (Photo 1). Titania sol was made from alkoxide of titanium. After coating this on glass substrate by dip coating method, it was dried and sintered. By repeating this process, we were able to manufacture TiO_2 thin film photocatalyst that was transparent and had excellent durability and high performance.

This TiO_2 fixed photocatalyst has surface composed entirely of TiO_2 so it can efficiently break down chemical substances that come in contact. After fixing the TiO_2 thin film photocatalyst onto transparent glass substrate, light that passes through the substrate can be used, and water treatment can be done continuously and without maintenance. Moreover, it has antibacterial and superhydrophilic properties.

Photo 1. Functional glasswares with photocatalyst coated with TiO_2 transparent thin film.

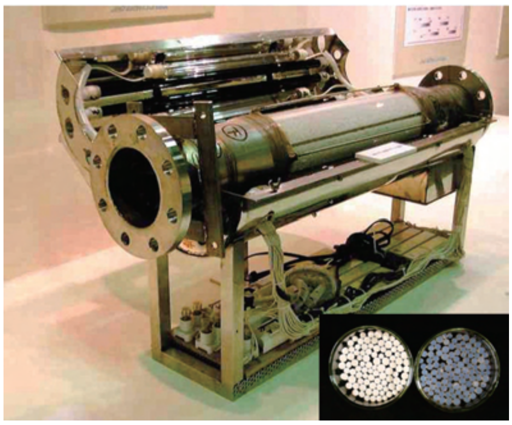

Photo 2. Photocatalytic silica gel (right, left is ordinary silica gel) and dioxin decomposing apparatus.

It is necessary to apply even and thin coat of titania sol on the substrate to manufacture high-performance TiO_2 thin film photocatalyst with excellent transparency and durability. Application with brush produced brush lines, or thin and thick areas that resulted in clouded film. Therefore we employed dip coating. However, in dip coating, when the speed of withdraw was too fast, the film became thick resulting in cloud and brittleness. The film thickness became uneven unless the withdrawal was smooth, and distortion occurred when sintering, and the layer dissociated. Therefore it was necessary to withdraw slowly and smoothly. Since there was no suitable dip coating device on the market, we jointly developed the device with a company in Aichi Prefecture and succeeded in manufacturing TiO_2 transparent thin film photocatalyst.

We conducted further investigation for the application of this TiO_2 transparent thin film photocatalyst, and developed photocatalyst glassware and photocatalyst pellets to purify water, and photocatalyst fluorescent lamp with antifouling, deodorant, and antibacterial functions.

4.1.2 Development of TiO_2 transparent porous photocatalyst

Since photocatalysis is surface reaction, if photocatalyst could be fixed in porous material with large surface area such as adsorbent, the target substance can be attracted by adsorption and then decomposed efficiently by photocatalysis. However, since ordinary porous material is not transparent to light, and reaction does not take place when the photocatalyst is located behind the porous material. Therefore, to increase the function of photocatalyst, we considered using inexpensive, transparent, and porous silica gel as matrix, and developed photocatalyst coated with TiO_2 transparent thin film.

In photocatalytic silica gel, the fine pores within silica gel are coated with TiO_2 film. It can effectively decompose odor and treat water since it has specific surface area of 450 m^2/g and light can penetrate into the fine pores. Moreover, absorbed harmful chemical substances are broken down safely into carbon dioxide. This porous photocatalyst can be called self-regenerating adsorbent.

Photo 3. Antifouling effect on surface of photocatalytic permeable block (right).

This photocatalytic silica gel had been patented and commercialized by companies. Using this, we developed water treatment and exhaust gas treatment apparatus that can efficiently degrade and remove over 99 % of dioxins in exhaust gas and wastewater from industrial waste incinerators (Photo 2)[5][6]. We also developed decolorization system for colored wastewater and photocatalytic human waste treatment system[7].

We developed photocatalytic permeation block with photocatalyst applied to the surface of porous concrete block[8] (Photo 3). This not only has antibacterial, antifungal, and antifouling effects, but also adsorbs, decomposes, and removes NOx and SOx in the air. It also can be used for heat island measures since it captures rainwater and surface temperature decreases by evaporation. Waste materials such as coal ash are used as matrix to lower cost. It is used as sound absorbing block of sound barriers, and is also installed in parking lots to purify exhaust gas and as heat island measures.

4.1.3 Development of photocatalyst that can be used in textiles and plastics[9]

Since textiles and plastics were degraded by photocatalysis when they were mixed with photocatalyst, it was impossible to apply them to textiles and plastics. We devised ways of coating the surface of TiO_2 with ceramics that had no photocatalytic activity and developed TiO_2 photocatalyst particle in form of muskmelon or kompeito (sea mine shaped sugar candy) (Fig. 2).

Muskmelon-form particle is made by coating the surface of TiO_2 with silica without photocatalytic activity like the net of a muskmelon. When it is mixed into textile or plastics, the silica without photocatalytic activity on the surface prevents contact of TiO_2 with textiles or plastic and decomposition is controlled. This particle can be adjusted by coating the surface of TiO_2 with thick coat of silica with even fine pores.

The *kompeito*-form particles is made by coating the surface of TiO_2 with apatite without photocatalytic activity, like the horn of *kompeito*. Apatite composes bones and teeth, and has excellent biocompatibility. This *kompeito*-form particle can be adjusted as apatite grows naturally, thereby saving

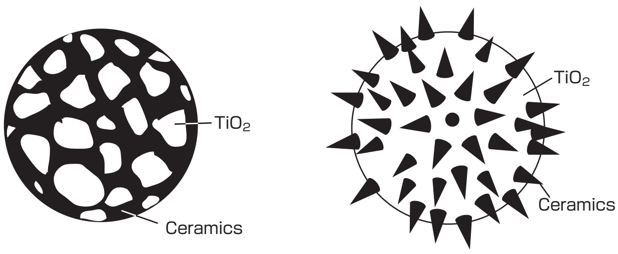

Fig. 2 Muskmelon-form and *kompeito*-form TiO_2 photocatalytic particles.

energy, on the surface of TiO_2 like growth of bone and teeth, by soaking the TiO_2 particle in solution of calcium and phosphate ions. Like muskmelon-form particles, when it is mixed in textiles and plastics, apatite without photocatalytic action on the surface prevents the decomposition of textiles and plastic by TiO_2. Since apatite can absorb bacteria and fungi, antibacterial and antifungal effects are obtained efficiently using this TiO_2 photocatalyst. The produced apatite is shaped like rose with large surface area, which enables great deodorant effect.

Both TiO_2 photocatalyst particles are patented by companies and are available commercially. Also, many textile and plastic products including curtain, men's suites, student's uniform, socks, mattress, towel, sheets, shoes, stuffed animals, artificial flowers (Photo 4), artificial plants, and wall papers are manufactured and sold in Japan and overseas, and practical application of photocatalysts is spreading dramatically.

By adding the *kompeito*-form TiO_2 photocatalyst particle to activated carbon, we developed functional adsorbent that can be used repeatedly. Active carbon can purify the environment by adsorbing harmful chemicals, odor, and VOC, but looses function when adsorption reaches saturation. When photocatalyst is added to active carbon, it adsorbs harmful chemicals, odor, and VOC without light, and photocatalyst breaks them down in presence of light, so environment can be purified efficiently. However, active carbon is also oxidized into carbon monoxide or carbon dioxide by photocatalysis. Therefore, we developed functional adsorbent that can be used repeatedly by controlling decomposition using *kompeito*-form TiO_2 photocatalytic particles. Active carbon has beautiful blue appearance and can be used as attractive interior piece with purification function (Photo 5).

Photo 4. Environment purifying artificial flower with *kompeito*-form TiO_2 photocatalytic particle.

We worked on various applications using this functional adsorbent, and had succeeded in agrichem-free cultivation of tomato by installing them in the ridge of the greenhouse[10][11]. By using functional adsorbent, the airborne bacteria and fungi pores in the greenhouse were reduced and disease and mold were prevented.

4.1.4 Compositing of photocatalyst and oxidant [12]

When wastewater is treated using photocatalyst, reaction time is high at first, slows down, and finally the reaction ceases. This is because dissolved oxygen is consumed when harmful substances in water undergo oxidative degradation, and it was found that oxygen was necessary to break down harmful substance by photocatalysis. Therefore, to prevent the cessation of photocatalysis, we tried to increase dissolved oxygen by aeration, and also composited photocatalyst and oxidant. When oxidants such as hydrogen peroxide and ozone are used with photocatalyst, the oxidants are converted efficiently to active oxygen by photocatalysis, and oxidative degradation is accelerated.

As application using this reaction, we developed dental bleach to whiten the teeth. This is a combination of TiO_2 and low concentration hydrogen peroxide, and teeth stain is broken down by applying to teeth and irradiating with light. By combining with hydrogen peroxide, the treatment could be done in short time. Teeth whitening used to be done with dangerous drug, but now it can be done safely with this method. When commercializing photocatalytic dental bleach, the Pharmaceutical Affairs Law was a barrier. After preparing safety data, it took three years to obtain approval of the Ministry of Health, Labour and Welfare, and the product was marketed on December 2006.

There is social valley of death for application to drugs, quasi-drugs, and medical equipment, and it is necessary to clear safety standards.

Further developing the photocatalytic dental bleach, we developed photocatalytic detergent for cleaning exterior walls. While antifouling and self-cleaning functions can be

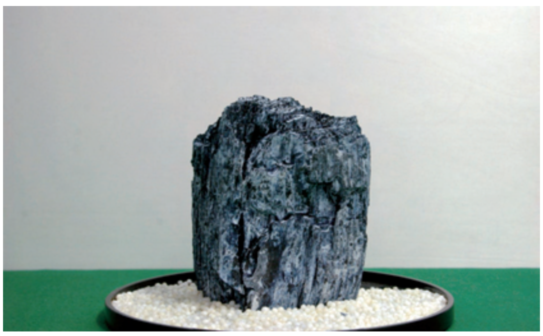

Photo 5. Photocatalytic functional adsorbent that is also attractive as interior piece.

obtained by coating the exterior walls with photocatalyst, the soil is decomposed by photocatalyst and flakes off along with it. Therefore, when applying photocatalyst, cleansing of substrate is extremely important. However, high-pressure water that is used generally requires large amount of water and may harm the exterior wall. Therefore, we developed a water solution of composited photocatalyst and oxidant. This was applied to the wall, left for some time, and then washed with sponge and water to remove the soil (Photo 6).

This photocatalytic detergent is safe and harmless, with only small amounts of TiO_2 and inorganic ions remaining after use. Moreover, when 300,000 bird flu viri were mixed with photocatalytic detergent, 99 % of them were deactivated 30 minutes later, and excellent effectiveness against bird flu was demonstrated. Also, we obtained deodorant effect along with antibacterial effect, and we developed deodorant and antibacterial apparatus and system using this detergent. It is beginning to be employed as deodorant and antibacterial measures in raw garbage treatment plant and nursing homes, as well as disease prevention in shrimp and fish farms.

We applied this photocatalytic detergent to water treatment by soaking the porous material in this detergent. Cost is an issue since large amount of purifier becomes necessary to purify waters of river and ocean. Aichi Prefecture, a major producer of Sanshu roof tile, disposes 380,000 ton/year of waste tile. Therefore, we developed low-cost photocatalytic water environment purifier using these tiles as matrix. We soaked the safe and harmless pellets made by grinding the roof tiles in photocatalytic detergent, and dried them. When these were dispersed onto the sludge in river or ocean, the sludge was decomposed and purified. This photocatalytic detergent has low cost at less than 100 yen per 1 kg, and use is expected in developing nations.

4.1.5 Development of visible light photocatalyst and new materials

While TiO_2 photocatalysts have many advantages as mentioned above, it will not function unless irradiated with ultraviolet light that has great energy. There is only 3~4 % ultraviolet light in sunlight, and much lower in fluorescent light. Therefore, to use photocatalyst efficiently indoors, photocatalyst that works under visible light is necessary. Currently, photocatalyst that works under visible light includes oxygen-defective and nitrogen-doped TiO_2 photocatalysts and those that use rare metal, but they cannot be used readily due to high cost.

Therefore, we developed low-cost visible light photocatalyst by combining TiO_2 and iron, which is low cost, safe, and harmless[13]. The cost is one-third of conventional visible light photocatalyst, and it is expected to become used widely mainly for indoor use.

Photocatalyst can be applied to antibacterial, antifungal, and freshness maintenance applications, and freshness and quality maintenance can be improved further when deoxidation function is added. Therefore, we developed TiO_2 deoxidant where oxygen was removed from TiO_2 by improving oxygen-defective TiO_2 photocatalyst[14].

As shown in Photo 7, this photocatalyst is blue and returns to white TiO_2 when it takes in oxygen, so it can be used as oxygen indicator. Unlike conventional iron deoxidant, there are several advantages such as it does not become red when mixed with food, does not combust in microwave oven, and does not sound the metal detector since it is not magnetic. This deoxidant is totally new application of TiO_2, and has advantages unseen in conventional deoxidants.

4.2 Collaboration of industry, academia, and government and standardization of testing method for photocatalytic performance

When developing high-performance photocatalyst by trial and error, cooperation of researchers and technologists in various fields such as catalyst science, material engineering, synthetic chemistry, analytical chemistry, applied chemistry, chemical engineering, and reaction engineering are necessary for quick and efficient investigation. Cooperation of companies with excellent production technology is necessary to realize photocatalyst.

In the Chubu region which is Japan’s industrial capital, there

Photo 6. Result of cleansing exterior wall using photocatalytic detergent.

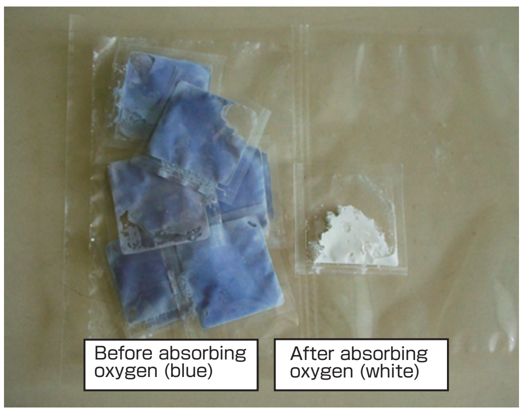

Photo 7. TiO_2 deoxidant before absorbing oxygen (blue) and after absorption (white).

are many companies, universities, and public testing research institutes with excellent capacity for R&D. Therefore, we decided to develop high-performance photocatalyst strategically utilizing and collaborating with their advanced production technology and R&D ability. We transmitted information actively through exhibitions, seminars, newspaper, magazines, and television. To seek collaborative R&D for photocatalysts, we also encouraged participation of researchers and technologists of various companies, universities, and public testing research institutes by holding research seminars for collaboration of industry, academia, and government.

This research seminar was the first photocatalyst industry group in Japan with about 350 members. It developed into the Society of Industrial Technology for Photocatalytic Articles (SITPA), the largest organization for photocatalysis with collaboration of industry, academia, and government, and was succeeded by the Photocatalysis Industry Association of Japan (PIAJ). We worked on setting quality standard for photocatalytic products, standardization of evaluation testing of photocatalytic performance, establishment of SITPA mark, as well as establishment of standard for labeling and terminologies. Our objectives were to prevent proliferation of poor products, to increase confidence for photocatalytic products, and to promote wholesome development of the photocatalysis industry. Moreover we worked on Japanese standardization (Japan Industrial Standard) and international standardization (International Organization for Standardization) for evaluation testing of photocatalytic materials, we organized and participated in international exhibitions of photocatalysts, and worked on education and diffusion of photocatalytic technology. This gathered interest of researchers, technologists, companies, and consumers, which in turn promoted new entries and increased number of cooperating researchers, technologists, and companies. Every year, we conducted 40 to 50 joint researches and technological advising for photocatalysts with various companies.

For evaluation of photocatalytic performance, following standards were adopted as Japan Industrial Standard:

- Fine ceramics (advanced ceramics, advanced technical ceramics) - Test method for air purification performance of photocatalytic materials - Part 1: Removal of nitric oxide (JIS R1701-1)
- Test method for antibacterial activity of photocatalytic products under photoirradiation and efficacy (JIS R1702)
- Test method for self-cleaning performance of photocatalytic materials - Part 1: Measurement of water contact angle (JIS R1703-1) and Part 2: Decomposition of wet methylene blue (JIS R1703-2)
- Test method for water-purification performance of photocatalytic materials by measurement of forming ability of active oxygen (JIS R1704)
- Light source for test of photocatalytic materials used under ultraviolet (JIS R1709)

Of these, testing of nitric oxide purification performance was established as ISO.

5 Results and discussions

We conducted development of high-performance photocatalysts that were unavailable before, and worked on various new applications. As result, about 200 patents on photocatalysts were filed in Japan and overseas, and about half of them are registered as patent. About 40 patents and property rights are in effect, and various products are manufactured and sold[15].

Although photocatalysts can be applied in various places, it is necessary to find optimal form and design for each application at product realization level. For example, for self-cleaning and antifouling, contamination is readily removed if the surface of the photocatalyst is smooth, while for water treatment and deodorization, uneven porous surface will absorb more harmful chemical substances. There are many kinds of odors such as acid, neutral, and alkaline, and photocatalysts to match each type are necessary.

By keeping specific use of the products in mind, we successfully developed various high-performance photocatalysts and photocatalytic products for specific purposes, and developed environmental purification technology using photocatalysis including water treatment, deodorization, air purification, self-cleaning, antifouling, antifogging, antibacteria/fungi, and dioxin treatment. Since the issue of cost is the greatest concern in commercialization, we worked to lower cost by using waste material and energy conserving manufacture method. As result, we realized practical use and commercialization of photocatalysts and environmental purification to some degree. However, achievement of goal of global purification is still ahead, and further research and diffusion of products are necessary.

6 Future issues

Photocatalyst can be used easily and safety, and it can be used anywhere in the world as long as there is light. Therefore, it can be used in developing nations as well as advanced nations, and it is a scientific technology that can contribute greatly to the world.

To realize purification of the earth environment, diffusion of photocatalyst in various countries around the world is necessary. Therefore, it is necessary to collaborate with research institutes and companies of East Asian and Southeast Asian countries where environmental pollution have become extremely serious due to rapid economic growth, to develop photocatalytic technologies corresponding to the situations of individual countries. For example, in areas where concentration of environmental pollutants is high, performance or surface area of photocatalyst must be increased or combined with other technologies. Also, cost reduction is necessary for diffusion of photocatalyst, and it is necessary to utilize waste materials and unused resource of that country. If we can develop a photocatalyst that matches the country, new application using it progresses and photocatalytic technology develops further.

Currently, associations for photocatalysts are established in China, Taiwan, and Korea and commercialization and diffusion of photocatalytic technology is progressing. However, to spread this technology to the rest of the world, further collaboration with technologists of various countries is necessary. We are working actively on cooperation and advising for research institutes and companies of various countries such as China, Taiwan, Korea, Thailand, Philippines, Vietnam, Europe and others.

Acknowledgement

For this research and development, I am grateful for the cooperation and support from researchers and technologists, companies, universities, public testing research institutes in Japan and overseas.

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Discussion with Reviewers

1 The progression of R&D

Comment (Hisao Ichijo)

I think the selection and integration of elemental technology is difficult to understand. The process by which R&D progresses while moving back and forth between *Type 1* and *Type 2 Basic Researches* is important. To enhance understanding of readers outside the specialty, the part that corresponds to *Type 1 Basic Research* should be explained briefly by citing original papers.

Answer (Hiroshi Taoda)

When developing catalysts and new drugs, there are support system to raise probability for how to raise effectiveness and performance by adding what, but basically it is done by trial and error. It is also a research for discovery and is *Type 1 Basic Research*. In general, technological development is thought to progress linearly from *Type 1* to *Type 2 Basic Research* and then to development and realization. However, it doesn't always happen that the knowledge (elemental technology) of *Type 1 Basic Research* are arranged neatly on the kitchen counter, and you can simply put them together and cook up a dish (*Type 2 Basic Research*). *Type 1 Basic Research* is done by trial and error, and the results are used to conduct *Type 2 Basic Research* and development/realization, and then *Type 1 Basic Researc*h is conducted again by feeding back the results. It is necessary to circulate *Type 1*, *Type 2*, and realization, and I believe this is *Full Research*. How I conducted research and thinking behind them were explained as much as possible in the individual sub-sections under chapter 4. Also, I added patents in the Reference to clarify the part that corresponds to *Type 1 Basic Research*.

2 The three " valleys of death"

Comment (Yoshiro Ohwadano)

I think it is interesting view that there were three kinds of "valleys of death" of technological, economic, and social valleys before photocatalytic technology could diffuse into the society. I think the whole article will become easier to understand if you provide description along this structure.

Specifically, please explain (1) where was the "technological valley of death" and how it was overcome, (2) where was the "economic valley of death" and how it was overcome, and (3) where was the "social valley of death" and how it was overcome.

This will sublimate the article to general knowledge that can be applied to other themes, and the article will become valuable.

Answer (Hiroshi Taoda)

(1) Technological valley of death was the fact that powder of photocatalyst was difficult to handle and photocatalytic performance was low. We overcame these issues by devising method for increasing effective surface area by fixing the photocatalyst onto transparent porous matrix. We also found ways to prevent decomposition of matrix, and improved performance by widening the sensitivity to visible light range. The function of photocatalysts increased significantly, and use and handling became easier.

(2) Economic valley of death was that the competitive power was lower compared to existing technology in terms of cost. We overcame this by cost reduction and seeking original usages. We used inexpensive base material such as waste material, and devised ways of fixing photocatalyst on transparent base and textiles.

(3) Social valley of death was legal regulations and low social acceptance. We overcame this by taking time to obtain approval. We also established performance evaluation method and worked on JIS and ISO standardization by organizing technological association under cooperation of industry, academia, and government. We also worked on education and diffusion of photocatalytic technology by appealing appropriate use of photocatalysts.

Is an angular standard necessary for rotary encoders?

— Development of a rotary encoder that enables visualization of angle deviation —

Tsukasa Watanabe

[Translation from *Synthesiology*, Vol.1, No.4, p.296-304 (2008)]

A rotary encoder is an angular readout device that reads an optical pattern written on the circumference of a shaft similar to a 360 degrees protractor and outputs angle position information. Since the output of a rotary encoder deviates from the true angle position due to optical pattern errors or eccentricity of the shaft about the rotation axis, users have encountered difficulty in ensuring the reliability of the angle information derived from an encoder. In order to solve this problem, “SelfA: self-calibratable angle device” was developed. The SelfA can detect a variety of angle deviation sources by itself, and has the ability to output an angular calibration value. This SelfA can detect the angle deviation value from an optical encoder which has been a black box until now and enables “visualization” of these factors quantitatively.

Keywords : Rotary encoder, angle standard, deviation evaluation, self-calibration

1 Introduction

A rotary encoder is a device to measure angles, and is used in machine industry for machine tool and semiconductor manufacturing as well as for angle measurement and control of industrial robots. It enables complex and precise manufacturing by adding the freedom of polar coordinates for “angle” to Cartesian coordinates composed of “length.” Office printers are equipped with rotary encoder as mechanism for accurate paper feed to enable precise printing. Rotary encoder is used as measurement device for “angle” in wide-ranging field from advanced measurement to familiar office appliances. Users are now demanding higher resolution, smaller size, and higher function. Manufacturing companies have tried to meet user demand by creating products with various forms and functions such as absolute and incremental encoders, magnetic and optical encoders, and modular, hollow shaft, and shaft type encoders, and ones with or without bearing. Product with resolution of over 0.1″ (1″ = 1º /3600 = approximately 5 μrad) had been fabricated.

Recently, users started to demand reliability of angle information obtained from the rotary encoder. In controlling the arm of a robot, smooth movement can be achieved by increasing the number of scale tick marks written on the 360º /cycle and thereby increasing the resolution. To control the position of the arm accurately, deviation between the angle information provided by the rotary encoder and the ideal angle position is evaluated, and accurate angle position control can be achieved by correcting the angle deviation. To accomplish this, it is necessary to measure the factor that cause the deviation of angle information obtained from the rotary encoder as shown in Fig. 1 and Fig. 2. However, although encoder manufacturers possessed technology to measure deviation in several tens of graduated scale, they did not have the technology to measure several thousands to ten thousands of scale, and therefore could not comprehensively and quantitatively evaluate various factors of angle deviation. As result, “accuracy” described in the product catalog of the encoder was not calibration curve that represents the angle deviation as shown in Fig. 3, but instead, it was safety tolerance where angle deviation is set as 0 and upper and lower lines are used to block off the large area of safety tolerance. Therefore, the “accuracy” as defined by the manufacturer was “black box” value where which angle deviation factors are included in what values were unknown.

Inherent deviation factor
(factors that arise from internal structure of encoder)
Deviation from scale
Eccentricity of encoder shaft and scale disc
Acquired deviation factor
(factors that arise due to usage by user)
Mechanical structural factor
Eccentricity of encoder shaft and device shaft
Distortion at time of attachment
Shaft wobble, bearing, etc.
Measurement environment factor
Temperature difference, secular distortion, etc.

Fig.1 Deviation factors of angle information.

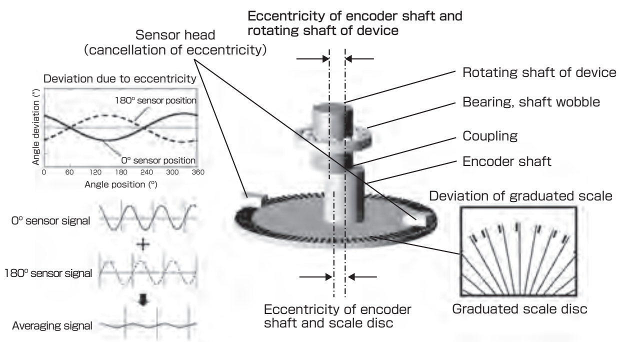

Fig.2 Concept of angle deviation factor of rotary encoder.

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Users were forced to place confidence in this "accuracy" when they used the encoder, and had no clue for how to investigate angle deviation in the angle information, and did not know how to maintain reliability.

To maintain reliability of angle measurement demanded by users, other than the manufacturers' effort in decreasing angle deviation through technological developments such as increasing precise and rigidity of the parts, breakthrough was necessary for new technological element that allowed measurement of angle deviation that could be done by users themselves and therefore improving reliability. "Visualization" of angle deviation as described in this paper is this breakthrough.

The National Institute of Advanced Industrial Science and Technology (AIST) has been conducting R&D of high-accuracy measurement device to establish the system of traceability for providing measurement standard. Development of National Standard for Angle[1] was started in 1997. Currently, the originally developed angle calibration device can measure the angle deviation as calibration values of several hundred thousand graduated scales of rotary encoder in short time. With uncertainty of calibration value at 0.01″, it is a national standard with highest accuracy in the world. This paper describes our work on calibration technology for angle deviation by self-calibration method and the development of self-calibratable rotary encoder SelfA[2][3]. I shall also explain the methodology of the research.

2 Angle deviation and current situation

First, what are the angle deviations in the angle signals output by rotary encoder? The cause of angle deviation of rotary encoder can be roughly divided into inherent and acquired factors, as shown in Figs. 1 and 2.

Inherent factor of deviation is mainly caused by the structural defect of the rotary encoder itself, and is determined at the stage of manufacturing. This includes deviation factor of graduated scale and eccentricity factor of encoder shaft and graduated disc. The scale deviation factor is angle deviation caused because the actual graduated scale does not correspond to the ideal graduated scale position with equal angle intervals as shown in Fig. 2 (bottom right). The eccentricity factor of encoder shaft and disc is angle deviation caused by the gap between the center of rotating shaft of rotary encoder and center of the scale disc.

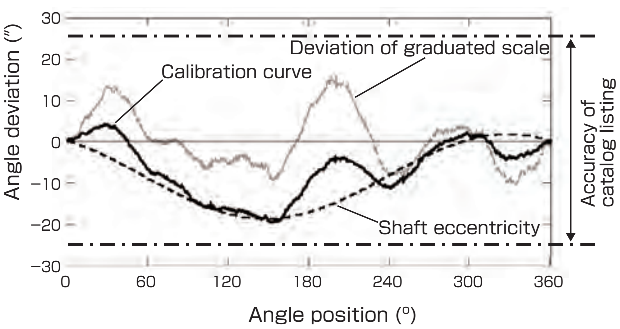

Fig. 3 Example of deviation factors in calibration curve.

Acquired factor of deviation is caused when the user attaches the rotary encoder to the device used or occurs during use. This may be caused by eccentricity of attachment of encoder shaft and rotating shaft of device, distortion of encoder during attachment, or distortion of encoder transferred from distortion of device due to temperature change during use. Moreover, shaft wobble dependent on the quality of bearing of device shaft may behave like motion eccentricity and cause angle deviation.

The eccentric errors between shaft-disc and shaft-shaft mentioned for inherent and acquired deviation factors are output as deviation represented as 1-period sine function as shown in Fig. 3.

To achieve high accuracy in rotary encoder, following technological methods were employed to keep the inherent and acquired deviation factors as small as possible.

(1)Improvement of quality of tick marks of graduated scale (spacing at equal angle interval, linearity of tick marks, etc.)
(2)Reduction of eccentricity by improving function of attachment jig and coupling
(3)Reduction of distortion by using robust and rigid parts to counter temperature change
(4)Cancellation technology for shaft eccentricity using 2 sensor heads

The items in (1)~(3) are methods for reducing the fundamental factors of angle deviation by increasing the preciseness of the parts of the rotary encoder. In contrast, cancellation technology for shaft eccentricity using two sensor heads is a method to cancel deviation caused by eccentricity in real time, as shown in Fig. 2 (left middle), by taking the average of signals measured by sensor heads that are accurately positioned at 180º opposite of each other on the graduated scale. As shown in Fig. 2 (left bottom), the sensor heads generate sine voltage signals for each interval between the graduated scales. If there is eccentricity, phase difference is produced in the sine voltage signals output by the sensor heads at 0º and 180º positions, and eccentricity can be cancelled by adding the two voltages. However, when the magnitude of eccentricity surpasses 1/4 of scale interval, or when the positioning accuracy of the sensor head surpasses 1/4 of scale interval, the voltage value may be reduced and the signal may not be output. For example, if the scale interval is 20 μm, shaft eccentricity and positioning accuracy of sensor head must be within 5 μm, so high preciseness of the parts is necessary as in (1)~(3). Therefore, there is method of installing several sensor heads[4], but in practice, it is difficult to position four or more sensor heads due to problem of positioning accuracy.

3 Research scenario

Since the only information available to the user was "accuracy" according to "black box" catalog, the user must use the encoder installed in the device with doubt, since whether the angle deviation, which may be caused by shaft eccentricity of attachment and change in environment are within the tolerance range of catalog "accuracy," is unknown. However, high accuracy measurement and control are possible by achieving several times higher accuracy compared to catalog "accuracy" by correcting the angle signal using the calibration value, if "visualization" of angle deviation can be achieved by calculating the calibration value (curve) of the rotary encoder as shown in Fig. 3.

The National Standard for Angle developed by AIST can measure angle deviation of graduated scale for several hundred thousand markings of rotary encoder. This enables quantitative evaluation of inherent and acquired deviation factors such as measurement environment, as shown in Fig. 1. However, almost all other acquired deviation factors vary and change according to individual difference of encoder, attachment, and environment used. Therefore, it is important for users to be able to "visualize" by measuring the calibration value when the encoder is installed in the device actually used. Therefore, we decided to achieve "visualization" of angle deviation by adding self-calibration function where the rotary encoder could measure the angle deviation and output the calibration value.

4 Self-calibratable rotary encoder "SelfA"

As shown in Table 1, several principles of self-calibration method[5]-[7] for measuring the angle deviation of rotary encoder have been devised. In the National Standard for Angle, the method for simultaneously recording the angle deviation of both encoders was employed by conducting self-calibration between the reference rotary encoder inside the National Standard and the user's rotary encoder that was the subject of calibration, using EDA-method (equally divided average method) [8]. Therefore, we developed the National Standard type small calibration device[9] that enabled self-calibration by EDA-method between the two encoders by installing separate encoder to the rotary encoder used by the user, as in the National Standard. However, it was difficult to obtain space for installing small National Standard around the rotary encoder that was already installed in the device used by the user, and we gave this up since we felt the limitation of downsizing.

Next, we investigated the principle of applying self-calibration using one rotary encoder. Multiple reproducing head method[10][11] and 3-point method[12] are methods in which several sensor heads are installed around the disc to measure the Fourier component by considering the angle deviation as consecutive 360º periodic curve as shown in Fig. 3. Using one of several sensor heads installed as standard, sensor heads are installed at 180º, 90º, 45º, 22.5º, etc. in case of multiple reproducing head method. In 3-point method, two places are selected from the arrangement of the multiple reproducing head method. This is a method where Fourier component can be obtained from the arrangement of other sensor heads against the standard sensor head, and calibration curve is obtained by inverse Fourier transformation. Therefore, accuracy of Fourier component that can be measured is greatly influenced by individual differences and accuracy of the sensor heads. Therefore, much labor is necessary for realization, and this method was not developed further.

Therefore, we decided to devise a method for EDA-method for user's rotary encoder by extending the EDA-method that was employed in the National Standard. Fig. 4(a) is the schematic diagram of EDA-method. Self-calibration is accomplished between the reference rotary encoder in the device where several sensor heads are installed at equally divided intervals around the disc, as shown on bottom, and the user's rotary encoder that is to be calibrated, as shown on the top. Fig. 4(b) shows that EDA-method can be done with one encoder by installing sensor head that will be standard among the sensor heads arranged at equally divided intervals. In Fig. 4(c), EDA-method is accomplished by using one of the sensors arranged at equally divided intervals as standard sensor head in Fig. 4(b).

4.1 Principle of SelfA

The EDA-method where self-calibration can be done with one rotary encoder is explained briefly. When five sensor heads are arranged around the disc of the rotary encoder as shown in Fig. 4(c), the angle signals produced by each sensor

Table 1. Principle of calibration for rotary encoder.

Calibration method	Cross calibration method	EDA-method	Multiple reproducing head method	2- and 3-point method	Divider method
Number of encoders	2		1		
Characteristic	Whole group comparison method	Fourier component measurement method			Sequential measurement method
Advantage	High accuracy	High accuracy, short time	High accuracy, short time	Short time	Short time
Disadvantage	Long time, labor intensive	2 encoders	Effect of individual head difference	Effect of head individuality Head interval must be exact	Accumulation of uncertainty

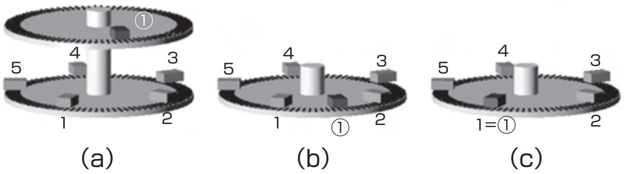

Fig.4 Transformation of EDA-method.

head include angle deviations A_1, A_2, A_3, A_4, and A_5. However, since each sensor head measures the same disc, A_2, A_3, A_4,and A_5 are phase shifted by 72º intervals against A_1. Since angle deviations cannot be directly separated from the angle signals, by calculating the difference δ between the angle signal of the first sensor head that has been set as standard, difference δ can be expressed only by angle deviation, as in equation (1). The example of measurement values of δ for 360º cycle is shown in Fig. 5.

$$\begin{aligned}\delta_1&=A_1-A_1\\ \delta_2&=A_1-A_2\\ \delta_3&=A_1-A_3\\ \delta_4&=A_1-A_4\\ \delta_5&=A_1-A_5\end{aligned} \tag{1}$$

Next, when the average value μ of the five δ is calculated, following equation is obtained. Fig. 6 shows the result when the average value μ of the five δ in Fig. 5 is calculated.

$$\mu=\frac{1}{5}\sum_{j=1}^{5}\delta_j=A_1-\frac{1}{5}\left(A_1+A_2+A_3+A_4+A_5\right) \tag{2}$$

The first term of right side is angle deviation of rotary encoder or the calibration value, but the average μ of the analyzed value cannot be called calibration value as is, due to the presence of second term. To check the relationship between first and second terms, the second term is calculated by creating A_2, A_3, A_4, and A_5 that are shifted by 72º phases, assuming that the calibration value of A_1 is obtained. Then the Fourier component has the relationship as shown in Fig. 7. It can be seen that the second term on right side is equal to the 5th order component of Fourier component of A_1. That is, when five sensor heads are installed, average μ is the calibration value of A_1 that does not include the 5th order component according to second term. When the effect of 5th order component on the calibration value is great, calibration value of higher accuracy can be obtained by arranging different number of sensor heads, for example seven. Since the calibration values obtained is measurement of specific Fourier component, this principle is categorized as method for Fourier component measurement as shown in Table 1.

The characteristic of this principle is that there is very little influence on angle deviation even if there is some shift in sensor head arrangement, because in the aforementioned multiple reproducing head and 3-point methods, statistic accuracy can be increased by calculating each sensor head that are arranged at equal angle intervals as standard, rather than relying on the relationship of one standard sensor head against all other sensor heads. Also, analysis can be done by four arithmetic operations without using Fourier transformation or inverse Fourier transformation.

4.2 Application of SelfA

As shown in Fig. 8, we developed a rotary table equipped with self-calibratable rotary encoder where 10 sensor heads are arranged on the bottom. Figure 9 shows the calibration

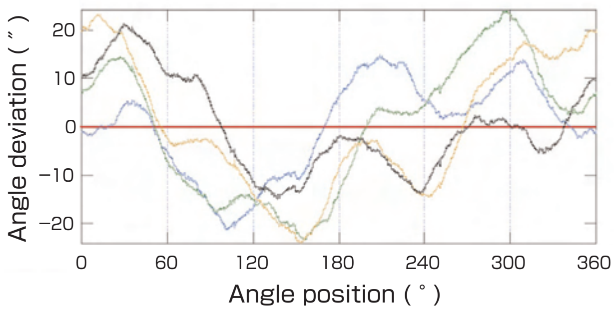

Fig. 5 Data output from SelfA.

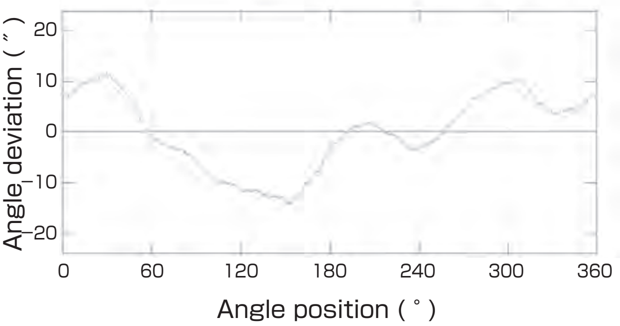

Fig. 6 Analysis result of SelfA.

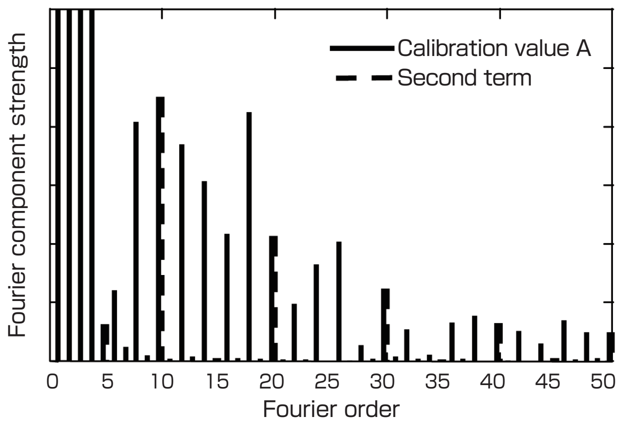

Fig. 7 Analysis result and relationship with Fourier component.

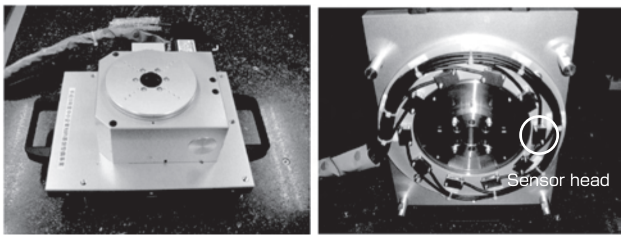

Fig. 8 Rotating table equipped with self-calibratable rotary encoder “ SelfA”.

values when self-calibration was conducted without anything on the top of table, and when 5 kg weight was placed for recalibration. It can be seen that the calibration values were apparently different. This is because the load distorted the table, and as result deformed the rotary encoder. The correction of angle deviation can be done at high accuracy by obtaining new calibration values.

Fig. 10 is a graph of data obtained for the calibration value of each rotation when the rotary table was turned 10 times. Unless calibration values are obtained, the catalog "accuracy" can only be expressed as ± 10″, but highly precise angle position of ± 0.3″, which is reproduction of calibration value, can be obtained. Fig. 11 is a graph that shows the reproducibility (variations) from the average value of the 10 calibration values shown in Fig. 10. In fact, it is known that the cause of ± 0.3″ variation is the non-reproducibility of the rotation of internal ball of the ball bearings in the rotary table.

The self-calibratable rotary encoder SelfA was found capable of measuring and outputting the calibration values for the eccentric factor of encoder shaft and device shaft that could not be measured until now, factor of distortion during attachment, and factor of measurement environment factor, as well as inherent deviation factors including the deviation factor in disc and eccentric factors of encoder shaft and disc.

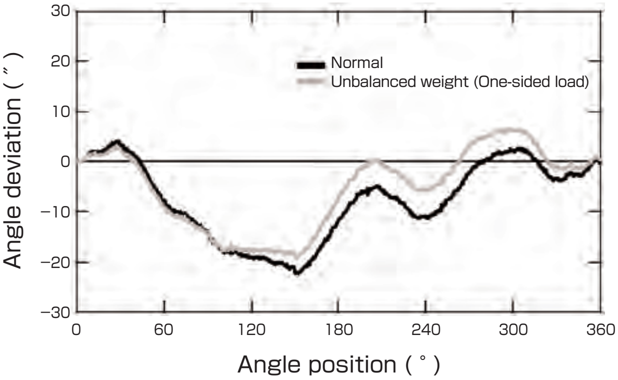

Fig. 9 Change in calibration value of internal rotary encoder caused by load.

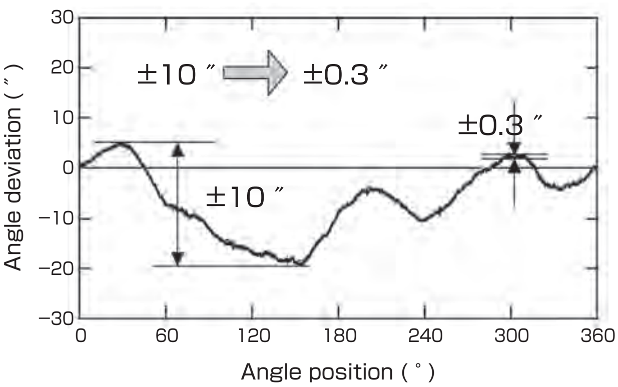

Fig. 10 Calibration value of rotary table (static angle deviation).

5 Future scenario

The self-calibratable rotary encoder SelfA described above can be called a rotary encoder that enables "visualization" of angle deviation and high accuracy through self-calibration methodology, which is different from conventional attempt of seeking higher preciseness of parts. For rotary encoders currently available commercially, the performance of product with highest accuracy is about ± 0.2″. However, SelfA has ability to evaluate the performance of ball bearings as shown in Fig. 11, and maintains quantitative reproducibility of ± 0.3″ upon measuring attachment deviation factor, measurement environment factor, mechanical structure factor, etc. If air bearing that has less shaft wobble than ball bearing is used, it can measure angle deviation at reproducibility surpassing ± 0.1″. Also, as the application example shows, SelfA not only measures angle deviation of the rotary encoder, but can be applied as new sensor for items other than for angle, such as rigidity of the device against the withstand load, shaft wobble evaluation, and bearing quality assessment, by using the "visualization" characteristic of angle deviation. This methodology can be applied to higher precise hardware technology to provide higher reliability to users.

For example, using SelfA's function that enables measurement of various angle deviation factors, highly precise measurement and control can be done in various sites by replacing with conventional rotary encoders, for angle control of radiotelescope and surveying instruments (total station and theodolite[Term 1]) used outdoors where the temperature difference is severe, gonio table such as x-ray device and elipsometer[Term 2] with offset load, angle control of machine tools and industrial robot that are subject to exterior force such as processing pressure and torque variation, and

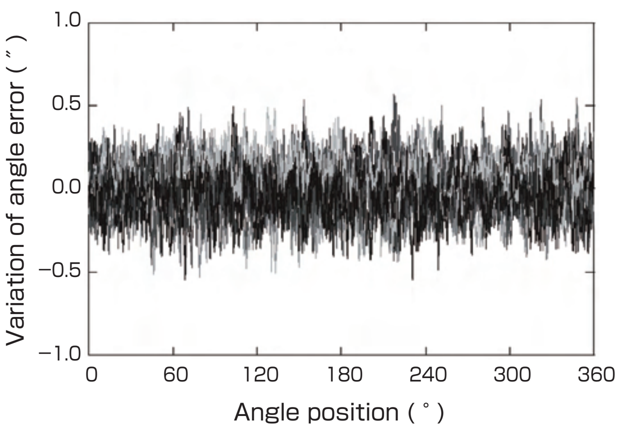

Fig. 11 Reproducibility of calibration value of rotating table (dynamic angle deviation).

accuracy evaluation of bearing used.

To realize this, development of SelfA in various sizes is necessary so the users can replace the inbuilt rotary encoder with SelfA described here without altering their current devices.

6 Know-how for practical application

Here, I present the necessary technological know-hows in utilizing self-calibratable rotary encoder SelfA.

6.1 Number of sensor heads

As indicated in Fig. 7, the Fourier component dependent on the number of sensor head cannot be obtained from the angle deviation obtained from the principle of SelfA. For example, when five sensor heads are installed, one cannot obtain 5th order Fourier component as 5, 10, 15…. If there are six sensor heads, the component for 6, 12, 18… cannot be obtained. Also, the Fourier component for angle deviation tends to become smaller at higher order in general. Therefore, if the number of sensor head installed is increased, the highly influential low order component can be measured, and one can obtain calibration value with no gaps in Fourier component to high order. Fig. 12 shows the relationship of number of sensor heads and achievable accuracy. For example if the achievable accuracy is 0.1″, 10~15 sensors are necessary, but five is sufficient for 1″. Therefore, it is not necessary to use so many sensor heads depending on the accuracy desired by the user, and optimal number of sensor heads can be selected.

6.2 Arrangement preciseness of sensor head

In conventional rotary encoder, two sensor heads had to be installed with strict limitation of 1/4 or less of scale interval to cancel the angle deviation by eccentricity. Moreover, shaft eccentricity and scale deviation variation were limited to 1/4 or less of scale interval. SelfA requires installation of not just two, but several sensor heads on the disc at equal angle intervals, and one may question the actual realization of Self A. The answer is "yes." SelfA does not require real time analysis, because each sensor head records the measured angle signal of the scale position by computer, and the difference of equations (1) and (2) are calculated after the measurements are completed, and the measurements can be done even if there is angle deviation that surpass the scale interval by eccentricity. Also, the sensor head does not output angle signal for each and every scale tick marks, but outputs angle signal as average value of several tens or several hundreds tick marks as shown in Fig. 13. Therefore, the change of angle deviation between proximal angle signals is small, so there is no particularly great effect on calibration value if they are installed few ticks away.

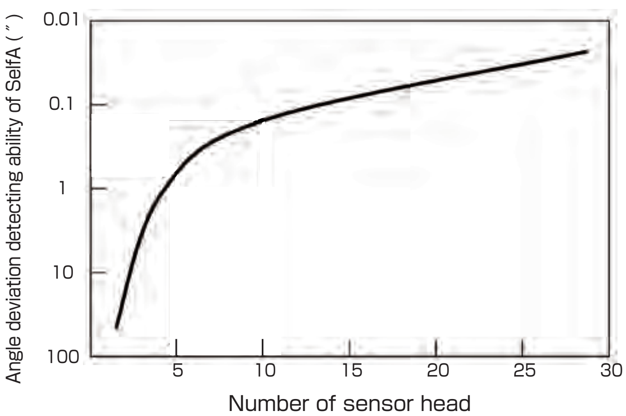

Fig. 12 Relationship between number of sensor head of SelfA and achieved accuracy.

7 Summary

The development of rotary encoders by companies was conducted under methodology of reducing angle deviation by employing precision parts or to erase the deviation with cancellation technology. However, development in this research employs visualization method by actively presenting all angle deviation. The companies that aimed for product with small angle deviation as possible and the stance of this research that aimed to evaluate angle deviation by development of National Standard for Angles enabled the development of the new self-calibratable rotary encoder SelfA.

The National Standard for Angle calibrates rotary encoder by EDA-method, which is one of self-calibration methodologies, rather than high accuracy angle standard. This means that any user or company can possess "angle standard" equivalent to the National Standard. Moreover, self-calibratable rotary encoder SelfA itself is capable of measuring angle deviation without calibrating device. This means that user or company may not need to own "angle standard." However, what becomes unnecessary is higher level "angle standard," and "angle standard" will become more familiar with the diffusion of self-calibratable rotary encoder SelfA.

As "things that cannot be measured cannot be made,"

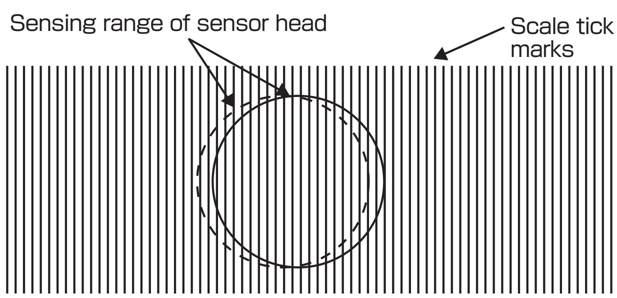

Fig. 13 Scale measurement size of rotary encoder.

establishment of high accuracy measurement technology is mandatory in manufacturing. “Visualization” of high accuracy status is thought to increase the reliability of accuracy evaluation that was done blindly until now, and is thought to contribute further for advanced manufacturing.

Terminology

Term 1. Total station,Thedolite: Theodolite is a surveying instrument to measure angle. It is optical device to measure the rotational angle in horizontal and vertical directions in triangulation. Total station is a theodolite with mechanism to measure distance to the target.

Term 2. Ellipsometer: Ellipsometery is measurement done by irradiating light onto sample and measuring the elliptic polarization of light reflected by sample. The device analyzes the optical constants such as thickness of film and refraction and coefficient of absorption. Rotary encoder is used to measure angle of reflection.

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Discussion with Reviewers

1 Aim of research and title

Question & comment (Motoyuki Akamatsu)

This research, in which the angle calibration technology for angle standard is developed into technology to maintain reliability of rotary encoder, is an excellent example of utilizing basic research in the society. The objective of this research is improvement of reliability, where the accuracy of encoder that is attached as a product can be calibrated frequently, and the accuracy can be maintained. However, I think the impact of introduction of this research result into the society, as “improvement of reliability” is difficult to understand to the general reader. How about adding explanation and emphasizing the social impact? Also, please reconsider the title and subtitle to emphasize the social impact.

Answer (Tsukasa Watanabe)

Although there are 100 years of history for length standard, and the world has developed national standards using same principle at same pace, there are only 20 years of history for angle standard. AIST started development of National Standard 10 years ago, but the principles for national standard around the world are diverse and not unified. Moreover, other countries increased preciseness of the parts to improve the accuracy of their national standards, and as result, the device became expensive and complex. This is the barrier that prevents common principle to this day. However, National Standard of AIST employs EDA-method as mentioned in this paper. Based on this principle, anyone can own device equivalent to National Standard.

This research started from making this principle more compact and creating a device that can be used inexpensively and simply by anyone. It is true that there was ambivalence and conflict in creating a standard while creating device that does not require standard.

Since the history of angle standard is still young, there are many black boxes such as what factors cause deviation in angle measurement devices and how such factors can be estimated. For “improvement of reliability,” we can obtain hint for opening the lid of the black box and thereby enabling quantitative evaluation unlike the “accuracy” described in the catalog, and both manufacturer and user can engage in discussion under same perspective on factors that cause angle deviation. I think that is truly standard for angle.

I drastically changed the title and subtitle as follows.

Development of self-calibratable rotary encoder
– Realization of improvement of angle accuracy and reliability of visualization of deviation factor

↓

Is an angular standard necessary for rotary encoders?
– Development of a rotary encoder that enables visualization of angle deviation

Question & comment (Mitsuru Tanaka)

In paragraph 2 of chapter 1, it is unclear why recent users are having trouble due to lack of reliability in accuracy. It is important to indicate what should have been the function desired by the encoder users, if it was not resolution. Also, I think it is good that you focused on situation "after installation" which is very important for the user. However, guarantee of reliability of angle value after installation to the customer's device is the job of users, so shouldn't it be, "Users are having trouble because they cannot guarantee the reliability of angle information obtained from the encoder after installation"?

Answer (Tsukasa Watanabe)

In the middle of chapter 1, I added explanation of reasons why the resolution and angle deviation became black box using the example of robot arm. As mentioned in chapter 2, I divided the inherent factors of angle deviation determined at the manufacturing stage of the rotary encoder, and the acquired factors of angle deviation determined when the user uses the rotary encoder. By doing so, I was able to explain the difference in catalog "accuracy" and the actual rotary encoder that are currently commercially available. The items with which the users are finding trouble can be specified, and I was able to provide explanation as you have indicated.

Therefore, I changed the expression to the following sentence: "Users were forced to place confidence in this "accuracy" when they used the encoder, and had no clue for how to investigate angle deviation in the angle information, and did not know how to maintain reliability."

2 "Visualization" as breakthrough

Question & comment (Motoyuki Akamatsu)

In paragraph 3, chapter 1, you write about the necessity of introducing new technology to improve reliability and also mention that "visualization" is necessary as breakthrough. Can you explain the scenario for selection of technology, such as whether "visualization" was mandatory for improvement of reliability, or whether "visualization" was selected over other options?

Answer (Tsukasa Watanabe)

There are several factors that cause angle deviation in angle signal output from the rotary encoder. I divided them as static and dynamic error factors, but I decided this categorization was not appropriate for the paper. The angle deviations generated by manufacturer and user are ultimately synthesized and expressed as inseparable angle deviation. Therefore, I changed the category into inherent and acquired factors of angle deviation. However, the most serious problem was that both manufacturer and user did not have ways of quantitatively evaluating angle deviation, and this lead to black boxing of angle deviation and decreased the reliability.

The companies used two hands, "increasing preciseness of parts" and "cancellation of deviation (eccentricity)" to keep the black box as small as possible. These two technologies will undoubtedly remain important technologies. However, the paper suggested a third hand, "self-calibration function" or "visualization."

I presented the reason for introducing "visualization" by expanding on chapter 2.

I wished there were better names for inherent and acquired factors of angle deviation.

Question & comment (Mitsuru Tanaka)

Dividing static and dynamic factors of angle error as in this paragraph should be part of elemental discussion in terms of methodological description. However, more you explain this division, I find it more difficult to understand the relationship between "conventional" and "visualization." Isn't there any way of making it clear? I think it will help to sort out the scheme of static = manufacturer/other-calibration = conventional and dynamic = user/self-calibration = visualization.

Answer (Tsukasa Watanabe)

I changed from categorization of "static and dynamic factors of angle error" to "inherent and acquired factors of angle deviation." I also explained (1)~(4) which are efforts by manufacturers to reduce the inherent factors of angle deviation.

Question & comment (Mitsuru Tanaka)

For research scenario, it can be said that this description is the essence of methodology. However, if it is so important for users themselves to "see," shouldn't you emphasize that? You say "visualization," but it is unclear about who is doing the seeing. Also, while it took lots of time, manpower, and cost to "see," now it became simple (that is, by self-calibration). Isn't this the development of methodology?

Answer (Tsukasa Watanabe)

The problem is that the manufacturer did not possess device to evaluate inherent angle deviation, and for users, all angle deviation were in black box so they had no idea how to take measures against the actual angle deviation or how to deal with catalog "accuracy." It was necessary to introduce technology where the users, as well as manufacturers, can measure angle deviation themselves to enable "visualization" of angle deviation. To do this, rather than evaluating the rotary encoder installed in the device, the rotary encoder itself could output angle deviation to conduct "visualization" of angle deviation easily and in short time, without requiring other device.

Question & comment (Mitsuru Tanaka)

The reader will be confused unless you explain the relationships among National Standard, encoder "made" by manufacturers, encoder "used" by users, and "encoder" that is calibrated in the research scenario. Also it is unclear why downsizing is necessary.

Answer (Tsukasa Watanabe)

I added the words "user," "National Standard," and "reference" in front of "rotary encoder" to clarify which rotary encoder was being discussed. Since the technology for "visualizing" angle deviation is complex, manufacturer and user become reluctant to use them when the device becomes large. Therefore, if the objectives are practical use and diffusion, it is necessary to allow "visualization" of angle deviation while it keeping the device almost same size as currently used rotary encoder. I specified the text as follows: "However, it was difficult to obtain space for installing small National Standard around the rotary encoder that was already installed in the device used by the user, and we gave this up since we felt the limitation of downsizing."

3 "Visualization" technology

Question & comment (Motoyuki Akamatsu)

It is argued that the point of this technological development is "visualization," but I think it is slightly different from the general meaning of "visualization," that is to understand the situation by visualizing the data. From the description of the paper, I understood that this development is about increasing accuracy by using the average values as calibration data, but if this is so, I don't think it is necessity to visualize. If you are using "visualization" as having different meaning from analysis based on visualizing, please add an explanation.

Answer (Tsukasa Watanabe)

Rotary encoder equipped with two or four sensor heads are marketed. However, they were used for cancellation of certain angle deviation such as shaft eccentricity. This is average operation in second term without first term on right side of equation (1). In fact, the slight difference in this equation plays role from "cancellation" to "visualization." Therefore, average is not calculation to raise statistical accuracy, but is used to calculate calibration value from rotary encoder itself without any standard. However, the effect is great, and we succeeded in visualizing angle deviation as calibration curve against "catalog accuracy" sandwiched between two lines as shown in Fig. 3.

Question & comment (Mitsuru Tanaka)

In the explanation for visualization technology, shouldn't you explain that "individual difference and installment accuracy of sensor head" was the lethal valley of death, and "distortion by temperature change and attachment" were valley of death that was overcome? Or else, it sounds like final result is that of things that were overcome.

Answer (Tsukasa Watanabe)

The methods of "National Standard (self-calibration using two rotary encoders)," "multiple reproducing head method and 3-point method," and "cancellation of eccentricity" all had lethal valleys of death against measurement of angle deviation such as "individual difference and installment accuracy of sensor head" and "distortion by temperature change and attachment." Although "downsizing of National Standard" was not lethal valley of death, size was valley of death. Therefore, whether EDA-method could be accomplished with one rotary encoder was a key.

4 Self-calibration function

Question & comment (Motoyuki Akamatsu)

From the explanation of Paragraph 3, Section 4, I understood that the feature of SelfA was to install several sensor heads at equal intervals within the rotary encoder, and one of them was used as standard sensor head. However, it looked same as EDA-method (bottom part of Fig. 4(a)) used in the National Standard. That is, the method in Fig. 4(c) looks the same as "installing several sensor heads around the scale disc" described in Paragraph 2. In the next sentence of Paragraph 2, difficulty of installing several sensor heads is described, but didn't difficulty of installing several sensor heads become problem in SelfA? For example, was this avoided by using equation (2)? Including these points, can you describe the process of finding the method used in SelfA? Also, Table 1 will become clearer if you describe the comparison of methods used in relation to the calibration principles shown in Table 1.

Answer (Tsukasa Watanabe)

The National Standard (bottom part of Fig. 4(a)) shown in Fig. 4, in fact, not only had five sensor heads, and although abbreviated in the figure, there was one more rotary encoder in the bottom, and the measurement of equation (1) was done separately while controlling one sensor head in five places in order of 1, 2, 3, 4, and 5. The lowest rotary encoder was selected so position control at 1/4 of the interval of the scale was possible, and that enables ideal measurement. As result, we achieved world's highest accuracy of uncertainty 0.01″.

However, because we developed this ideal device, the concept that "EDA-method is done using 2 rotary encoders" and "ideal sensor head installment" were fixed, we could not move on to the idea a→d→c of Fig. 4. However, by setting the target uncertainty to 1″, I think various ideas became possible. When the target was set at uncertainty of about 1″, we were able to consider analytic algorithm that was different from National Standard, like there isn't much effect on calibration curve even if the sensor head is slightly off. In cancellation technology, "1/4 of scale interval" is a must.

In multiple reproducing head and 3-point methods, the measurable Fourier component is determined by the position of other sensor heads against standard sensor heads. Therefore, the positional relationship of standard sensor head and other individual sensor heads is important. However, in EDA-method, they are arranged equiangularly and isotropically, so some degree of averaging occurs. Moreover it is extremely difficult to express them quantitatively.

Question & comment (Mitsuru Tanaka)

For description of calibration technology, the explanation "Fourier component dependent on the number of sensor head cannot be obtained" for Figure 7 and the one in the text are insufficient, and the reader cannot imagine the main content of the technology.

Answer (Tsukasa Watanabe)

In reference to Fig. 7, I added specific explanation: "For example, when five sensor heads are installed, one cannot obtain 5th order Fourier component as 5, 10, 15…. If there are six sensor heads, the component for 6, 12, 18… cannot be obtained. Also, the Fourier component for angle deviation tends to become smaller at higher order in general. Therefore, if the number of sensor head installed is increased, the highly influential low order component can be measured, and one can obtain calibration value with no gaps in Fourier component to high order."

5 Explanation of terminology and improvement of expression

Question & comment (Motoyuki Akamatsu, Mitsuru Tanaka)

Please add a section for terminology, since there are terms such as total station, theodolite, ellipsometer, and calibration, which are unfamiliar to people outside of the field. Particularly, the word "calibration" is closely related to the main theme of visualization of angle error, and should be explained properly. Also, the terms "error" and "accuracy" are used with bias of standard specialist, so they should be corrected.

Answer (Tsukasa Watanabe)

I added terminology section for total station, theodolite, and ellipsometer. I also revised the paper so it could be understood that the meaning of calibration is to calculate the deviation of angle as calibration value. For "error" and "accuracy," I changed the terms to "deviation" and catalog "accuracy."

Methodology and a discipline for synthetic research

— What is *Synthesiology*? —

Hideyuki Nakashima

[Translation from *Synthesiology*, Vol.1, No.4, p.305-313 (2008)]

In the analytical sciences, several formulations of the discipline such as Descartes' discourse, Kuhn's paradigm, and Popper's falsifiability have been attempted, but such discussion has not been done for *Synthesiology*. The only exception is the series of work by Yoshikawa. Here, I attempt a formulation of the methodology of *Synthesiology* as a discipline by stating that what Yoshikawa calls *Type 2 Basic Research* is synthetic research.

Keywords **:** Synthetic methodology, perspective, worldview, language, science, engineering

1 Introduction

Ever since Descartes' *Discourse de la Méthode*[1], methodologies for natural sciences have been discussed widely. Natural sciences demand objectivity, and Popper demanded "falsifiability"[2] to guarantee objectivity and stated that only propositions that can be falsified by experiment could be subjects of science. The necessary condition for this is that the system to be observed must not include any observer so that the same result can be obtained by any operator. This means science cannot exist if the system under investigation cannot be separated from the observer.

Kuhn's paradigm theory[3] suggested that the framework is not fixed but is changing as the paradigms shift, and Polanyi's theory of tacit knowledge[4] addressed the fact that natural science is a social activity. While these concepts lead to questions regarding the absolute status of natural science, they did not give rise to discussions on other methodologies, particularly in engineering. The only exception is the series of work by Yoshikawa[5]-[8].

The author learned natural science methodology in high school and university, and only recently became aware that it is not almighty[9]. To study only subjects to which scientific methodology can be applied is like searching for lost items only under were there is a bright light. There exist in this world many issues to which scientific methods fail to fit, and I wish to consider what methodologies can be employed to tackle such subjects. This is the main theme of *Synthesiology*. In the world of crafts, the artist and the work are inseparable (i.e. they do not fulfill the necessary condition of natural science), and in the field of engineering, though not totally dependent on people as in crafts, they cannot be completely separated as in pure science. Here, I shall state that what Yoshikawa calls *Type 2 Basic Research* is synthetic research, and attempt a formulation of methodology as a research discipline.

2 Language and thought

I shall consider synthetic methodology from the standpoint of a researcher of information science. Since I consider language to be the essence of issue, I wish to focus discussion on language.

First, I shall discuss *kagaku*. In English there is a word "science," but this does not correspond directly to Japanese *kagaku*. In English "science" and "art" are concepts that overlap as shown in Fig. 1. The origin of "science" is *scientia* (knowledge), and it started as a classification of the study subjects. The origin of "art" is ars (craft or craftsman's skill), and the nuance is close to Japanese *gijutsu* (commonly translated "technology"), and is a concept that includes *geijutsu* (commonly translated "fine arts"). In Japanese, *kagaku* and *geijutsu* do not overlap, and the two words are often used as antonyms. In Japanese, the part "science" minus "art" is generally called *kagaku*. Yoshikawa's *Type 1 Basic Research* corresponds to this part. The part where "science" and "art" overlap is the contact point of *kagaku* and *gijutsu*, or *kogaku* (commonly translated as "engineering").

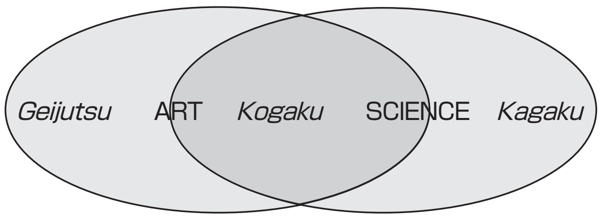

Fig. 1 *Kagaku* and *kogaku*.

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Yoshikawa's *Type 2 Basic Research* corresponds to this part. The part "art" minus "science" is called *geijutsu*. However, the word *kogaku* is rarely used as defined here, and in most cases it has come to mean *Product Realization Research* as described by Yoshikawa. The study of synthesis described in this paper is a methodology for the overlap of science and art.

Before discussing the main theme, I would like to explain the issue of language and thought more deeply.

There have been various studies on the effects of culture on cognition[10][11]; its effect on language was addressed by Sapir-Whorf's linguistic relativity hypothesis [12][13].

・Whorf's strong hypothesis (linguistic determinism): thought is determined by the language spoken by that person

・Whorf's weak hypothesis (linguistic relativity): categorization of concept differs by language and culture

Although these concepts have yet to be proven, I believe them to be basically correct (at least the theory of linguistic relativity).

Whorf claims that the setting of time and space is also determined by language[13].

Newtonian space, time, and matter are no intuitions. They are recepts from culture and language. That is where Newton got them.(Reference [13], p. 153)

We dissect nature along lines laid down by our native languages. The categories and types that we isolate from the world of phenomena we do not find there because they stare every observer in the face; on the contrary, the world is presented in a kaleidoscopic flux of impressions which has to be organized by our minds–and this means largely by the linguistic systems in our minds. We cut nature up, organize it into concepts, and ascribe significances as we do, largely because we are parties to an agreement to organize it in this way–an agreement that holds throughout our speech community and is codified in the patterns of our language. The agreement is, of course, an implicit and unstated one, but its terms are absolutely obligatory; we cannot talk at all except by subscribing to the organization and classification of data which the agreement decrees. (Reference [13], pp. 213-214)

Spatial cognition has not yet been set immediately after we were born as a baby, but the differences in such cognition gradually occur in the process of learning language such as Japanese or English. The most famous issue is the recognition of color; psychological studies show that how and where one categorizes color depends on the kinds of names of colors in one's mother tongue. Whorf's hypothesis attempts to broaden this to the concept of space in Newtonian mechanics.

Although not as radical as Whorf, it can be readily imagined that the structure of language influences on cognition, particularly on the scientific way of thought. Particularly relevant to this theory is that whether the world can be considered as a *mono* (thing) or perceived as an experience of *koto* (event). Bin Kimura[14] stated that when regarding an apple as a subject, or when a *mono* called an apple is viewed, it is objectified as something separate from oneself. However, when one describes the *koto* of a falling apple, the account includes the concept that the person is experiencing it. Perhaps being related to this, English syntax is noun-centric, while Japanese syntax is verb-centric [15]. It is reported that in Western languages, acquisition of nouns by children precedes acquisition of verbs, but in China, this is not the case, and in some cases they are acquired in the reverse order[16].

3 Perspective

Since the linguistic description is closely related to the perception of the world (positioning of oneself), I shall present an example that clearly demonstrates this case. Kanaya noticed the difference between the Japanese and English perspectives, and stated that the two languages describe the environment as follows:

・English has the perspective of a God

・Japanese has the perspective of an insect

Kanaya[17] discusses the experiment by Yoshihiko Ikegami in a "Series Japanese" shown on the NHK educational channel. Yasunari Kawabata's *Yukiguni* (*Snow Country*) starts with the sentence:

(1) *Kokkyo no nagai tonneru wo nukeruto yukiguni deatta.*

E.G. Seidensticker, a noted scholar and translator who worked on various Kawabata literatures, translated this into English as follows:

(2) The train came out of the long tunnel into the snow country.

When people who read the sentence were asked to draw this scene, people who read the Japanese sentence (1) drew from the perspective of a passenger on the train (Fig. 2), while those who read the English (2) drew a bird's eye view of a

Fig. 2 Perspective of insect.

train coming out of a tunnel (Fig. 3). The difference is not a result of a poor English translation. Although a description from the Japanese perspective is possible, it does not become a natural English sentence.

Next I would like to discuss the issue of the perspective in research. When studying a certain system, where does the researcher stand? In the natural sciences, the researcher is positioned in a place clearly separate from the system (Fig. 4 left). The perspective is that of external observer who stands and observes the system from the outside. Ideally, the behavior of the system should not be affected by the observation, or the method of removing the interference of the observation activity from the result is known. In contrast, Fig. 4 right shows the perspective of internal observer who is part of the system.

According to Ichikawa[18], Westerners enforce a consistent worldview where the world can be explained according to a consistent set of rules and it is assumed that there is an existence that transcends the system such as a God or a constitution (Fig. 4 left). On the contrary, Japanese presupposes a potentially-inconsistent worldview where different rules are accepted for different groups. It is interesting that there is a similar point as in the difference between English and Japanese languages. If thought is determined by language, does it not mean that the Japanese are an appropriate ethnic group to introduce a study of synthesis (*Synthesiology*) to the world?

I shall summarize the prerequisite of natural sciences:

- Analytical methodology can be applied
  - Observation does not interfere with the subject
  - If there is any interference, it can be calculated (including the quantum uncertainty principle)
- Objectivity can be maintained
  - The perspective of the external observer is maintained
  - The consistency of the world can be maintained

Fig. 3 Perspective of bird.

Although the perspective of the internal observer is not scientifically desirable, some fields must assume this situation. One must inevitably take internal perspective into account in creating/constructing a system. As will be explained later, in constructing a new system, the steps of first setting specifications, drafting a plan, and then implementing it do not necessarily flow smoothly. The phase of using and evaluating the created system is necessary, and in this case, the researcher is included in the system as a user. In that sense, methodologies for analytic sciences and synthetic sciences are different. Discussion on the synthetic methodology begins by correctly recognizing this point.

The following fields may necessitate synthetic methodology (as will be explained later, this does not imply that analytical methods are unnecessary):

- Complex systems
- System with macro-micro interaction (like economics)
- Multilayered systems (like humans)
- Amorphous systems, which is crystal-like locally, but are uneven globally
- Once-only, non-experimentable phenomena (such as the theory of universe, geology, evolution, history, and archaeology).

The above discussion suggests that not small number of fields necessitate the use of synthetic methodology.

4 The loop of synthesis

In general, analysis and synthesis are considered activities that go in opposite directions. Analysis divides the whole into parts, and studies the individual parts and their interrelationships. In contrast, synthesis assembles the whole from parts. This rather simplistic view is based on the image of disassembly and reassembly, but rarely are parts available without a shortage or an excess of them at the start of the synthetic process, that is, in prior to we know what we are assembling. Synthesis must start from the identification of the parts. Moreover, it is extremely difficult to identify the parts from whatever one wishes to synthesize and there is no algorithmic method.

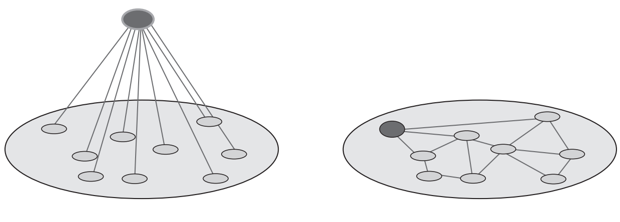


Fig. 4 External view (left) and internal view (right) of systems.

We think that the analytical method must be used as part of the synthetic method (Fig. 5). Take for example, architecture. When a requirement is given for a building, a building with functions matching to the requirement can be designed directly in an ideal case, but this is difficult unless the architectural style is standardized. Normally, something that fulfills the specifications is constructed (generated) (this is discussed further in Section 5). This construct may be a model or an actual house, but it is important to actually generate it. By actual generation, details that surpass the given requirement will always be added (specified), and unforeseen interactions with the environment may occur. Therefore it is necessary to analyze the construct and clarify its property. The procedure of analysis is not necessarily fixed and new procedure may be used after their generation. When analytical results are obtained, the necessary feedback is generated after comparison with the original specifications. The specifications may change in this process. The synthetic loop may not end here, and specifications may continue to change and be repeated. This loop is the core of synthetic method.

Notice that the specifications (goals) change within a synthetic loop. In the sense that an analytical procedure can be determined only after the actual construct is generated, analysis and synthesis are not simply activities in opposite directions, but can be considered to be orthogonal to each other.

When formulated as above, the creation of hypothesis in the natural sciences becomes a synthetic loop in the meta-level of theory formation. A hypothesis is generated, and an experiment (or a thought experiment) is designed to investigate the phenomenon that can be deduced from the hypothesis. The methodology for investigation of a hypothesis (i.e. an experiment) is well established in analytical sciences, while the method of evaluation and investigation of the products is not established for the case of synthetic methodology. I conjecture that the only synthetic evaluation methodology is a similar one to evaluation used for story-telling or novels (to be discussed later). In fact, in the evaluation of a hypothesis (which is a result of a meta-level synthesis), the "Ockham's razor" standard may be applied where simplest explanation is selected from multiple hypotheses that can explain the same phenomenon. This is perhaps an example of narrative evaluation.

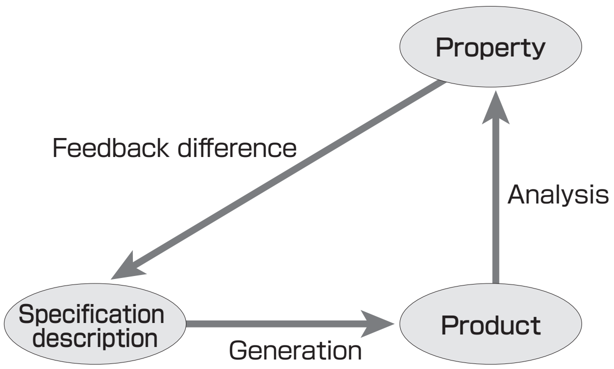


Fig. 5 Synthetic loop.

The synthetic loop may roughly correspond to the process of "*Type 2 Basic Research*" as described by Yoshikawa[8], and the aforementioned hypothesis creation corresponds to "abduction."

For example, the derivation of a principle in theoretical research is synthesis but its validity is verified by deductive analysis of its consistency with existing theory and by induction through experiments. For artifacts, this is verified by actual use in society. From this perspective, *Type 1 Basic Research* is totally different from *Type 2 Basic Research*. Considering the logical structures of *Types 1 and 2 researches*, they both include abduction, but the importance of abduction is greater for *Type 2 research* through all stages of the research process. Furthermore, in *Type 1 Basic Research*, the verification process is done by researchers themselves or by other researchers in the same discipline, but in *Type 2 Basic Research*, it is demonstrated in society, which is unrelated to the world in which research is conducted. (Reference [7], p. 6)

Since Yoshikawa considers *Product Realization Research*, he assumes the society is the only place of verification, but for arbitrary synthetic science, it is more suitable to consider the "environment" in general as a place of verification.

5 Evolutionary methodology

What is the methodology for "generation" in the synthetic loop? I believe the only possibility is a similar one to evolution. It is a search method commonly called "trial and error." To put it simply, evolution is the repetition of the following process (Fig. 6).

1. Various candidates are generated from existing seeds.
2. Candidates are evaluated, and only good ones are selected.

While the generation of candidates can be achieved mechanically, evaluation is more difficult in general. However, the various possibilities are not just generated randomly. Efficient search methods are necessary and the genetic algorithm is one example. Locally, hill-climb

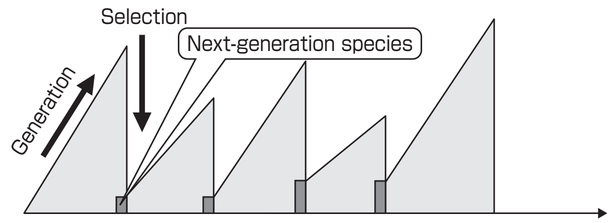


Fig. 6 Scheme of evolution.

algorithm or optimization method for parameters of equations may be applicable, but those are limited to areas where analysis of the subject has completed.

Ichikawa[18] sets the following conditions for a system to be an evolutionary system:

・Existence of self-replication unit (genome) to maintain regularity
・Existence of a system structure of self-replication units (existence of elements and a system that connects those elements)
・Possibility for mutation of the system structure
・Interaction (competition) among replicator systems (for frequency of replication)
・Existence of external environment

Ichikawa defines the scientific method as follows:

1. Prediction is made by deductive inference from a model that consists of hypothesis and constants.
2. Observation and measurements are planned and conducted to confirm this prediction.
3. A hypothesis is confirmed when facts obtained from observation and prediction match.
4. In case evidence is found that contradicts the prediction (a counterexample) is obtained, the hypothesis is rejected as false. Using the evidence, inductive inference is used to rebuild a new hypothesis. Return to 1.

Ichikawa claims that modern science and technology are comprised of an evolutionary system. This is evidence that evolutionary methodology is one of a synthetic method. On the other hand, it is difficult to argue that this is the only possible methodology. However, there are plenty of circumstantial evidences supporting the claim.

First of all, evolution in nature, or biological evolution, succeeds by employing this method.

Second, *shogi* (Japanese chess) and *go* that have thousands of years of history take this style. Only evolutionary methodology exists in these games, which more number of people who are as smart as or smarter than researchers had studied over the years.

There is no formula for win in *shogi* or *go*. Although certain well-analyzed series of moves are studied as *joseki* (the set sequence), other parts are just emergent or trial and error in form of *sakiyomi* or “reading ahead” where several future developments are evaluated sequentially. Also, explanation of the set sequence is provided in form of *sakiyomi*. In fact, there is no other method other than *sakiyomi*.

The process of generation discussed here is in the same direction as reductionism in analytical science in the sense that it involves generation of details (specific moves in *shogi*) to realize the property of whole (in *shogi*, goal of winning or of capturing the opponent’s pieces). From this perspective, I shall shift discussion to synthetic methodology to generation of a multilayered system.

6 Generation of a multilayered system

In this section, I shall elaborate on synthetic methodology with a focus on the generation of a multilayered system. First, I shall define a multilayered system.

It is necessary to understand the various layers listed below to understand the organism called a human.

・Society
・Individual
・Organs
・Cells
・Molecules

All these (conceptual) layers must be combined to understand a human. In analytical science, there is a method of explanation by isolating one layer at a time, but there is no methodology of understanding several layers together. It is impossible to understand human society only by molecular biology in a reductionism style. There is an individual law in each layer, and the lower layer is not a disassembly of the upper layer. That is, the existence of multilayer must be accepted as is.

I shall discuss the methodology for addressing the multilayer system. First, taking the analytical method, let us simplify our subject and consider two-layered system. I take as an example, the description of performing music according to Kimura[19] (Fig. 7). In playing music, there are two layers: the layer of the music that one wants to perform and the layer of the actual performance. Three factors enter the two layers.

1. A future noema[Term 1]: plan or the music score one wants to perform
2. A noesis: actual performance, actual notes played
3. A current noema: music conceived as the result of listening to notes played

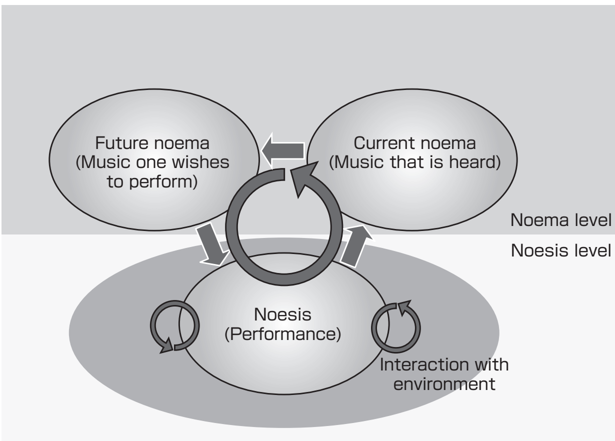


Fig. 7 Noema and noesis in musical performance.

Most important here is the interaction of noesis and the environment. In the example of music, temperature and humidity of the day, reverberation of the hall, response of the audience, and many more else are reflected in music. It is important to note that there are factors that cannot be controlled directly by the player. Almost all activities of generation involve such uncontrollable interactions. Traditionally, these were not considered important, but synthesis is very difficult because of the interaction with the environment. In case a company manufactures a product, for example, the user may use it in unexpected manner. Using pocket pager for message communication or explosive dissemination of mobile phones because of their unexpected usage are some examples. However, many in the fine arts actively employ this type of interaction. Bleeding and blotting of ink in shodo (Japanese calligraphy) and firing and ash inclusion in ceramics are good examples.

The product is analyzed after interaction with its environment. One becomes aware of how the music is being played, and the difference with what one actually had in mind is fed back to the next moment of performance. Continuing this loop is the synthetic method of musical performance, and it is a speedy loop with a high frequency. Research is carried out in a longer loop with a larger time constant. However, both loops have fractal structure, and similar loops can be observed when each transition is examined closely.

The reader may have become aware that the loop of noema and noesis has the same form as the aforementioned synthetic loop (Fig. 5). FNS diagram[Term 2] of synthetic methodology[20][21] (Fig. 8) shows this in chronological order.

The meanings of the arrows in Fig. 8 are as follows:
- (C1) Action conducted to realize the future noema.
- (C1.5) Generated noesis interacts with the environment.
- (C2) As a result, a current noema that is different from expected future noema is produced in the upper layer.
- (C3) Feedback action to a new future noema. This may include increasing the set of controlled variable and changing plans.

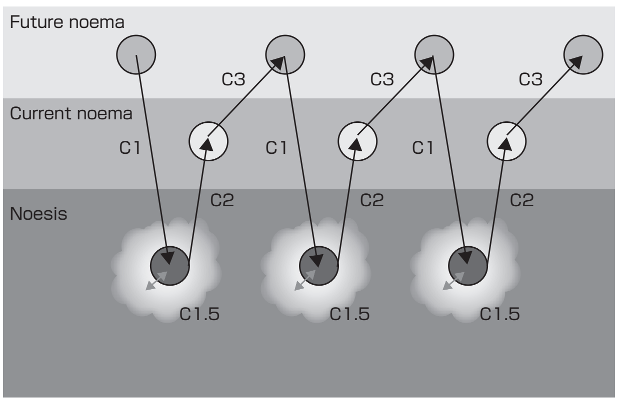


Fig. 8 FNS diagram of synthetic method.

The scheme of noema and noesis can be applied to the actions of a scientist engaging in natural science (analytical science) (Fig. 9). When the logic (or hypothesis) exists in the form of a future noema, then the apparatus for experiment is the externalization of the noema set up to justify the hypothesis. When the actual experiment is conducted, interactions with various factors in the environment take place to produce certain phenomenon (corresponding to a musical performance). Feedback is provided to the theory by analyzing the observed phenomenon (correction or justification).

When we extend the formalism to a multilayer system, the FNS expands to multilayer in the noesis level (Fig. 10)[22]. The figure shows three layers, and higher layers are on the left. The item that was an external environmental factor in the lower layer (right side) is internalized in the noesis in the upper layer (left side). That is, the system that consists of noesis (in the center) and other elements (distributed in the environment) on the lower layer (right side) become either the central noesis or one element of the environment in the upper layer (left side). For the example of music, the audience, who was included as part of the environment when seen from the layer of the player, becomes part of the system in the upper layer of the total performance. What is considered one system in the layer of the player is broken down into further subsystems (such as eyes, ears, or hands).

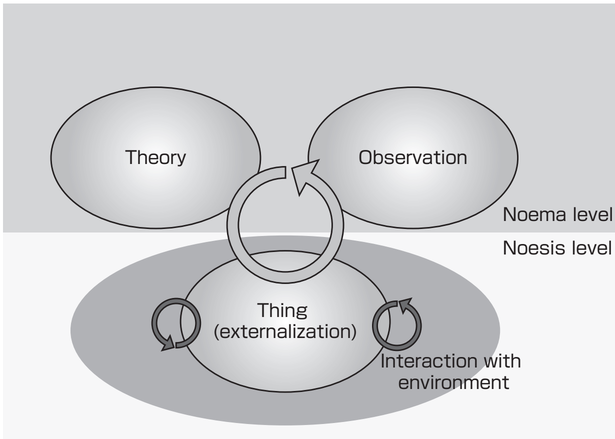


Fig. 9 Noema and noesis in act of science.

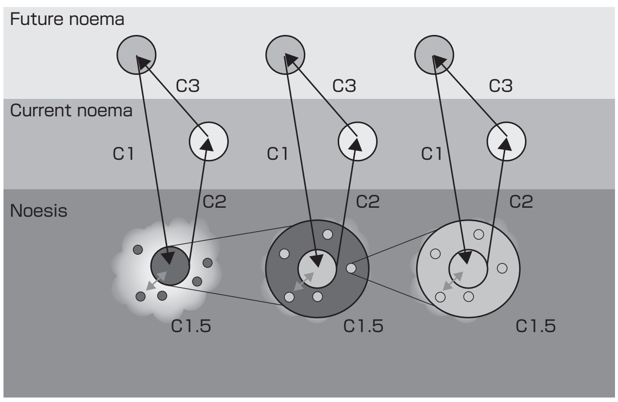


Fig. 10 FNS diagram for multilayer system.

In the lower level, noeses are decomposed along “part of” relationship. On the other hand, noemas take on a different description system. For example, individual level noema and cellular level noema form independent systems. Some of the relationship between different layers of noemas may be analyzable. The classic example is that temperature (upper layer) in thermodynamics is the average value of kinetic energy of molecules (lower layer), but it is rare that such relationship is known.

7 Narrative

I mentioned the hypothesis that the only method for evaluation or proof of synthesis is narrative methodology.

There is no index for evaluating a narrative objectively. Judgment of good or bad story is personal, and people do not necessarily agree. However, a good novel is accepted by many people and wins many awards. In that sense it may be possible to evaluate synthesis.

Good narratives often have the following conditions:

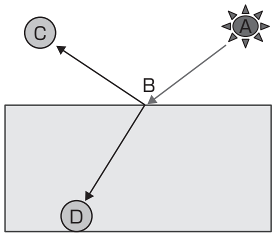
・ There is a strong relationship (causal relationship) among factors of a narrative

・ One factor should have relationships with as many other factors as possible (there should be no isolated factor not related to other factors).

・ It is better for the relationship between factors to be not too obvious.

Narrative explanation is sometimes used in physics. In fact there are two ways of explaining reflection and refraction (Fig. 11). One uses the law that the angle of incidence and the angle of reflection are equal, and the other is a teleological explanation that light travels via the shortest route from point A to point C. It is the same for refraction, where one can use either the angle of reflection or the shortest time.

The shortest time route cannot be determined unless the goal is known. Therefore, the route cannot be calculated for light itself. That is, the analytical explanation is a method in which time is eliminated and the argument is addressed as a spatial issue, but in a synthetic thinking along time, the laws of angles of reflection and incidence must be employed since they address a local mechanism (in fact, there are finer mechanisms of light as wave, but this will be not be discussed here since the essence remains the same)[23].



Analytical/teleonomic explanations	Synthetic explanation
"Shortest route" between AC (AD)	Law of angle of incidence and angle of reflection
Cannot be determined until position of C (D) is known	Projection of route (and C/D)

Fig. 11 Analytical (past) explanation and synthetic (future) projection.

8 Service engineering

The Center for Service Research was established at AIST, and service engineering also uses a synthetic discipline.

In a certain English-Japanese dictionary, there are 23 translations of word “service.” This means that 23 examples are introduced because there is no single concept (word) in Japanese corresponding to “service”. Therefore, the word “service” in “service engineering” can be interpreted differently by different people. I consider “service” to mean “to use” rather than “doing something free of charge.” Also, engineering is a synthetic methodology, the topic of this paper. Therefore, the service engineering is not a discipline of a tertiary industry service, but should be understood as related to a practical application of a synthetic discipline. The process of service engineering can be mapped onto the FNS diagram.

Figure 12 shows the direction of the future research that was summarized in the workshop[24] organized by the Japan Science and Technology Agency. In conventional R&D, the focus has been mostly on manufacturing things (bottom right), but the service part, in which the thing developed is actually used, is important. Continuation of the loop of use, evaluate, create new values if necessary, and then the return to R&D is “*Full Research*” in Yoshikawa’s term as I understand it, and synthetic methodology is covered in this diagram. In fact, when Fig. 12 is rotated by 120 degrees to left, it is mapped into the first cycle of FNS.

Peter Drucker foresaw the importance of use (service) in the 1960s, and following statements can be seen in *Age of Discontinuity*[25].

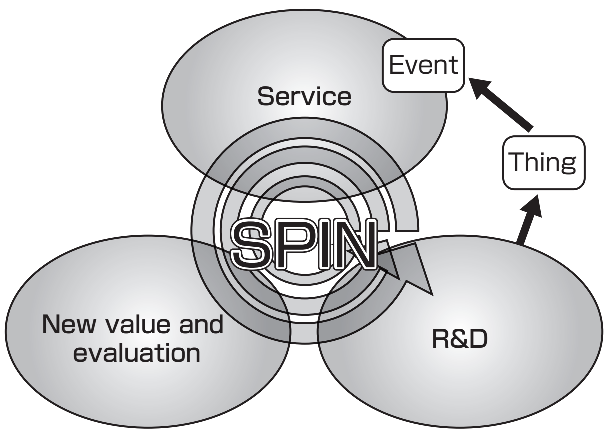


Fig. 12 State of new R&D including service in loop.

The search for knowledge, as well as the teaching thereof, has traditionally been dissociated from application. Both have been organized by subject, that is, according to what appeared to be the logic of knowledge itself. The faculties and departments of the university, its degrees, its specializations, indeed the entire organization of higher learning, have been subject-focused. They have been to use the language of the experts on organization, based upon "product," rather than on "market" or "end use." Now we are increasingly organizing knowledge and the search for it around areas of application rather than around the subject areas of disciplines. Interdisciplinary work has grown everywhere.

This is symptom of the shift in the meaning of knowledge from an end in itself to a resource, that is, a means to some result. Knowledge as the central energy of a modern society exists altogether in application and when it is put to work. Work, however, cannot be defined in terms of the disciplines. End results are interdisciplinary of necessity.

9 Summary

In the research and development there are many areas where the methodology of the natural sciences cannot be applied. I would like to emphasize this fact to the research community. The role of *Synthesiology* is to cover those realms. I have reviewed the methodology of synthetic disciplines in this article.

I stated that the structure of Japanese that we use, the perspective that is demanded by Japanese language, and thus the Japanese worldview is close to synthetic methodology. I attempted formulation of synthetic methodology.

Finally, I shall summarize the difference of the worldviews.

Consistent worldview	Potentially-inconsistent worldview
Monotheism	**Polytheism**
Cartesian dichotomous world	**Inseparable "色即是空 (form itself is emptiness)"**
Analytical method = science	**Synthetic method**
Objectivity (*mono* = thing)	**Subjectivity (*koto* = event)**

Formulation of synthetic method is an area where Japan can contribute greatly, and I think this is the major role of *Synthesiology*.

Terminology

Term1. Terms "noema" and "noesis" were originally conceived by Edmund Husserl, and Kimura uses them with slightly different meaning. Since it is very difficult to explain them simply, please refer to the source, or consider them as mere symbols in this article. I shall present alternative way of reading them, but these are not definitions of the terms. A noema is like a concept or plan. "Specification description" in Fig. 5 is an example of noema. A noesis is actualization of noema. The product of "Generation" in Fig. 5 is an example of a noesis.

Term2. Historically it was Fujii-Nakashima-Suwa diagram. Recently it was renamed Future Noema Synthesis diagram.

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Editorial Policy

Synthesiology Editorial Board

Objective of the journal

The objective of *Synthesiology* is to publish papers that address the integration of scientific knowledge or how to combine individual elemental technologies and scientific findings to enable the utilization in society of research and development efforts. The authors of the papers are researchers and engineers, and the papers are documents that describe, using “scientific words”, the process and the product of research which tries to introduce the results of research to society. In conventional academic journals, papers describe scientific findings and technological results as facts (i.e. factual knowledge), but in *Synthesiology*, papers are the description of “the knowledge of what ought to be done” to make use of the findings and results for society. Our aim is to establish methodology for utilizing scientific research result and to seek general principles for this activity by accumulating this knowledge in a journal form. Also, we hope that the readers of *Synthesiology* will obtain ways and directions to transfer their research results to society.

Content of paper

The content of the research paper should be the description of the result and the process of research and development aimed to be delivered to society. The paper should state the goal of research, and what values the goal will create for society (Items 1 and 2, described in the Table). Then, the process (the scenario) of how to select the elemental technologies, necessary to achieve the goal, how to integrate them, should be described. There should also be a description of what new elemental technologies are required to solve a certain social issue, and how these technologies are selected and integrated (Item 3). We expect that the contents will reveal specific knowledge only available to researchers actually involved in the research. That is, rather than describing the combination of elemental technologies as consequences, the description should include the reasons why the elemental technologies are selected, and the reasons why new methods are introduced (Item 4). For example, the reasons may be: because the manufacturing method in the laboratory was insufficient for industrial application; applicability was not broad enough to stimulate sufficient user demand rather than improved accuracy; or because there are limits due to current regulations. The academic details of the individual elemental technology should be provided by citing published papers, and only the important points can be described. There should be description of how these elemental technologies are related to each other, what are the problems that must be resolved in the integration process, and how they are solved (Item 5). Finally, there should be descriptions of how closely the goals are achieved by the products and the results obtained in research and development, and what subjects are left to be accomplished in the future (Item 6).

Subject of research and development

Since the journal aims to seek methodology for utilizing the products of research and development, there are no limitations on the field of research and development. Rather, the aim is to discover general principles regardless of field, by gathering papers on wide-ranging fields of science and technology. Therefore, it is necessary for authors to offer description that can be understood by researchers who are not specialists, but the content should be of sufficient quality that is acceptable to fellow researchers.

Research and development are not limited to those areas for which the products have already been introduced into society, but research and development conducted for the purpose of future delivery to society should also be included.

For innovations that have been introduced to society, commercial success is not a requirement. Notwithstanding there should be descriptions of the process of how the technologies are integrated taking into account the introduction to society, rather than describing merely the practical realization process.

Peer review

There shall be a peer review process for *Synthesiology*, as in other conventional academic journals. However, peer review process of *Synthesiology* is different from other journals. While conventional academic journals emphasize evidential matters such as correctness of proof or the reproducibility of results, this journal emphasizes the rationality of integration of elemental technologies, the clarity of criteria for selecting elemental technologies, and overall efficacy and adequacy (peer review criteria is described in the Table).

In general, the quality of papers published in academic journals is determined by a peer review process. The peer review of this journal evaluates whether the process and rationale necessary for introducing the product of research and development to society are described sufficiently well.

In other words, the role of the peer reviewers is to see whether the facts necessary to be known to understand the process of introducing the research finding to society are written out; peer reviewers will judge the adequacy of the description of what readers want to know as reader representatives.

In ordinary academic journals, peer reviewers are anonymous for reasons of fairness and the process is kept secret. That is because fairness is considered important in maintaining the quality in established academic journals that describe factual knowledge. On the other hand, the format, content, manner of text, and criteria have not been established for papers that describe the knowledge of "what ought to be done." Therefore, the peer review process for this journal will not be kept secret but will be open. Important discussions pertaining to the content of a paper, may arise in the process of exchanges with the peer reviewers and they will also be published. Moreover, the vision or desires of the author that cannot be included in the main text will be presented in the exchanges. The quality of the journal will be guaranteed by making the peer review process transparent and by disclosing the review process that leads to publication.

Disclosure of the peer review process is expected to indicate what points authors should focus upon when they contribute to this journal. The names of peer reviewers will be published since the papers are completed by the joint effort of the authors and reviewers in the establishment of the new paper format for *Synthesiology*.

References

As mentioned before, the description of individual elemental technology should be presented as citation of papers published in other academic journals. Also, for elemental technologies that are comprehensively combined, papers that describe advantages and disadvantages of each elemental technology can be used as references. After many papers are accumulated through this journal, authors are recommended to cite papers published in this journal that present similar procedure about the selection of elemental technologies and the introduction to society. This will contribute in establishing a general principle of methodology.

Types of articles published

Synthesiology should be composed of general overviews such as opening statements, research papers, and editorials. The Editorial Board, in principle, should commission overviews. Research papers are description of content and the process of research and development conducted by the researchers themselves, and will be published after the peer review process is complete. Editorials are expository articles for science and technology that aim to increase utilization by society, and can be any content that will be useful to readers of *Synthesiology*. Overviews and editorials will be examined by the Editorial Board as to whether their content is suitable for the journal. Entries of research papers and editorials are accepted from Japan and overseas. Manuscripts may be written in Japanese or English.

Required items and peer review criteria (January 2008)

	Item	Requirement	Peer Review Criteria
1	Research goal	Describe research goal ("product" or researcher's vision).	Research goal is described clearly.
2	Relationship of research goal and the society	Describe relationship of research goal and the society, or its value for the society.	Relationship of research goal and the society is rationally described.
3	Scenario	Describe the scenario or hypothesis to achieve research goal with "scientific words".	Scenario or hypothesis is rationally described.
4	Selection of elemental technology(ies)	Describe the elemental technology(ies) selected to achieve the research goal. Also describe why the particular elemental technology(ies) was/were selected.	Elemental technology(ies) is/are clearly described. Reason for selecting the elemental technology(ies) is rationally described.
5	Relationship and integration of elemental technologies	Describe how the selected elemental technologies are related to each other, and how the research goal was achieved by composing and integrating the elements, with "scientific words".	Mutual relationship and integration of elemental technologies are rationally described with "scientific words".
6	Evaluation of result and future development	Provide self-evaluation on the degree of achievement of research goal. Indicate future research development based on the presented research.	Degree of achievement of research goal and future research direction are objectively and rationally described.
7	Originality	Do not describe the same content published previously in other research papers.	There is no description of the same content published in other research papers.

Instructions for Authors

Synthesiology Editorial Board
Established December 26, 2007
Revised June 18, 2008
Revised October 24, 2008

1 Types of contributions

Research papers or editorials should be submitted to the Editorial Board.

2 Qualification of contributors

There are no limitations regarding author affiliation or discipline as long as the content of the submitted article meets the editorial policy of *Synthesiology*, however, authorship should be clearly stated. (It should be clearly stated that all authors have made essential contributions to the paper.)

3 Manuscripts

3.1 General

1) Articles may be submitted in Japanese or English.
Accepted articles will be published in *Synthesiology* (ISSN 1882-6229) in the language they were submitted in. All articles will also be published in *Synthesiology - English edition* (ISSN 1883-0978). The English edition will be distributed throughout the world approximately four months after the original *Synthesiology* issue is published. Articles written in English will be published in English in both the original *Synthesiology* as well as the English edition. Authors who write articles for *Synthesiology* in Japanese will be asked to provide English translations for the English edition of the journal.
2) The manuscript shall be prepared using word processors or similar devices, and printed on A4-size portrait (vertical) sheets of paper. The category of article (research paper or editorial) shall be stated clearly on the cover sheet.

3.2 Structure

1) The manuscript should include a title (including subtitle), abstract, the name(s) of author(s), institution/contact, main text, and keywords (about 5 words).
2) Title, abstract, name of author(s), and institution/contact shall be provided.
3) The length of the manuscript shall be, about 6 printed pages including figures, tables, and photographs.
4) The title should be about 10-20 Japanese characters (5-10 English words), and readily understandable for a diverse readership background. Research papers shall have subtitles of about 15-25 Japanese characters (7-15 English words) to help recognition by specialists.
5) The abstract should include the thoughts behind the integration of technological elements and the reason for their selection as well as the scenario for utilizing the research results in the society.
6) The abstract should be 300 Japanese characters or less (125 English words). The Japanese abstract may be omitted in the English edition.
7) The main text should be about 9,000 Japanese characters (3,400 English words).
8) The article submitted should be accompanied by profiles of all authors, of about 200 Japanese characters (75 English words) for each author. The essential contribution of each author to the paper should also be included. Confirm that all persons who have made essential contribution to the paper are included.
9) Discussion with reviewers regarding the research paper content shall be done openly with names of reviewers disclosed, and the Editorial Board will edit the highlights of the review process to about 3,000 Japanese characters (1,200 English words) or a maximum of 2 pages. The edited discussion will be attached to the main body of the paper as a part of the article.
10) If there are reprinted figures, graphs or citations from other papers, permission for citation, if needed, should be clearly stated and the sources should be listed in the reference list. All verbatim quotations should be placed in quotation marks or marked clearly within the paper.

3.3 Format

1) The text should be in formal style. The section and subsection chapters should be enumerated. There should be one line space at the start of paragraph.
2) Figures, tables, and photographs should be enumerated. They should have a title and an explanation (about 20-40 Japanese characters or 10-20 English words), and the position in the text should be clearly indicated.
3) For figures, clear originals that can be used for printing or image files (resolution 350 dpi or higher) should be submitted. In principle, the final print will be 15 cm x 15 cm or smaller, in black and white.
4) For photographs, clear prints (color accepted) or image files should be submitted. Image files should specify file types: tiff, jpeg, pdf, etc. explicitly (resolution 350 dpi or

higher) . In principle, the final print will be 7.2 cm x 7.2 cm or smaller, in black and white.

5) References should be listed in order of citation in the main text.

Journal – [No.] Author(s): Title of article, *Title of journal*, Volume(Issue), Starting page-Ending page (Year of publication).

Book – [No.] Author(s): *Title of book*, Starting page-Ending page, Publisher, Place of Publication (Year of publication).

4 Submission

One printed copy or electronic file of manuscript with a checklist attached should be submitted to the following address:

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The submitted article will not be returned.

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Proofreading by author(s) of articles after typesetting is complete will be done once. In principle, only correction of printing errors are allowed in the proofreading stage.

6 Responsibility

The author(s) will be solely responsible for the content of the contributed article.

7 Copyright

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Readers' Forum

Dear Editor

Please accept my sincere thanks for providing me issues 1 and 2 of *Synthesiology*. My reaction, I'm really impressed. This is a great undertaking and I wish you success in promoting the journal.

I read President Yoshikawa's introduction to the journal. Hopefully you will accomplish what he outlines and all the efforts in managing technology have not really achieved. The research you refer to as Type 2 Basic Research is certainly needed and your description defines the approach very clearly:

> "A form of research that integrates the knowledge of different disciplines or creates new knowledge when necessary, and transforms a concept into artifacts (product or service) that can be recognized by society."

In issue 2 your interview with MIT Professor Lester was most interesting. Excellent questions and excellent thoughts! This is an excellent opportunity to link academia and industry. It is an opportunity to promote integration of disciplines. It should be an opportunity to return to broad-based engineering and technology and management education in academia.

Perhaps we have become too specialized even within the disciplines. I look back at the electrical and electronic engineering curriculum in the US where it has become very specialized. In some cases we graduate students in electrical engineering without even knowledge of how an electric motor functions. They have no significant understanding of mechanics, dynamics, fluid flow, thermo, heat transfer, and other basic studies in engineering.

The articles in these two issues provide valuable material to begin the discussion about integration.

Well I wish you and your colleague's success in promoting integration of knowledge from different disciplines. Will Synthesiology be available in the US?

Best regards,

Gus Gaynor

Gerard H. (Gus) Gaynor, IEEE Life Fellow
President, Technology Management Council

Letter from the editor

It is our great pleasure to hand you *Synthesiology-English edition* Volume 1, Number 4. It contains papers from diverse fields. I thank all of the people who were involved in creating this issue.

Of particular interest to us is the relationship of *Synthesiology* and the studies by Dr. Osamu Shimomura, the Nobel Laureate in Chemistry in 2008. As you may know, Dr. Shimomura clarified the luminescence in Aequorea Victoria and discovered the green fluorescent protein (GFP). Dr. Yoshihiro Ohmiya, one of the contributors of the papers in this issue, wrote in the postscript that Dr. Shimomura's research is a typical *Full Research* that was put into practice through *Type 2 Basic Research* and *Product Realization Research*. Ohmiya *et al*. have developed interest in the luminescence mechanism of firefly, and in the process of clarifying this mechanism, have realized the technology for detecting multiple gene expression using luminescent protein (luciferase). This is an excellent example where the practical use has progressed from the basic research of bioluminescence mechanism that was the main theme of the present Nobel Prize. It can be called a new *Type 2 Basic Research*– a synthetic research.

Dr. Hideyuki Nakajima contributed an article on the methodology of the synthetic research and discipline system. In this article, a very interesting point is raised about language and thought, and he discusses the importance of meaning of words in the synthetic method. Particularly, I find the following points very unique: "English has the perspective of God; Japanese has the perspective of an insect" and "If thought is determined by language, I believe the Japanese are an appropriate ethnic group to introduce a study of synthesis (*Synthesiology*) to the world."

In relation to Dr. Nakajima's discussion, I shall mention two points about "words" used in the papers in *Synthesiology*. First point is the "term." Different technical terms are used in different research and technology fields. The terms unique to a certain discipline are the proof of accumulation and systemization of knowledge in that particular discipline, and there is significance in the fact that the terminologies are different from those of other fields.

On the other hand, in the papers of *Synthesiology*, the research is described as a synthetic method of *Type 2 Basic Research* that transcends the methodologies of individual disciplines. Therefore, appearance of new terms that appropriately describe this methodology is necessary. Perhaps it may take some time, but I expect terms unique to synthetic method will gradually take form. It also means the concept of synthetic method will gradually become clear as the terms develop.

The second point about words is the "language." In publishing *Synthesiology*, there was a heated debate on whether to use Japanese or English as a language of the journal. In one way of thoughts, the papers should be published in English to be read widely and internationally. In another way, to write papers on *Type 2 Basic Researches* that have never been done before, it is important for Japanese researchers to describe them in Japanese, and the content should first be read in Japanese language by Japanese researchers and engineers.

The Editorial Board decided to emphasize the latter viewpoint, and the journal will be published in Japanese (papers submitted in English will be published in English). On the other hand, we also considered the former viewpoint and decided to publish *Synthesiology – English edition*. Although the publication of the English editions will be a few months behind the Japanese editions, I hope they will be read widely as much as the Japanese edition.

We will be delighted to receive active submissions from overseas researchers and engineers. If you have an opportunity, please, spread the word of *Synthesiology* to researchers and engineers.

Senior Executive Editor
Naoto Kobayashi

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