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High-value materials from incineration residues of burnable garbage

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Contributing to the SpaceWire international standard

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2018

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Highlights of the Papers in *Synthesiology*

Synthesiology is a journal that describes the objectives, specific scenarios, and procedures of research activities that attempt to utilize the results in society, in particular, the process of synthesis and integration of elemental technologies for practical application. To allow the readers to see the value of the papers in a glance, the highlights of the papers that characterize *Synthesiology* have been extracted.

Synthesiology **Editorial Board**

Research paper: Challenge towards synthesis of non-silica-based hybrid mesoporous materials
—Level of compositional design and control of mesoporous materials achieved so far—
Tatsuo KIMURA
This paper describes the course by which non-silica-based hybrid mesoporous materials were synthesized, which was considered difficult until now. The strategies include the selection of new starting materials and the search for synthetic routes that did not exist before. As a result, the extension of composition and structure of the mesoporous material was successfully achieved. It shows the importance of the integration of elemental technologies using sub-nanometer scale chemical species such as organic and metal species as tools.

Research paper: High-value materials from incineration residues of burnable garbage
—Production of silica with high specific surface area from “molten slag” and direct transformation of silica to basic raw material for silicon chemical industry—
Norihisa FUKAYA *et al.*
This paper discusses the technology for producing high-functional materials that are valuable not only in the venous industry, but also in the arterial industry, using molten slag that has negative value of being incineration residues of garbage. Rather than achieving high purity through simple sorting as done conventionally, it is innovative that synthesis is performed based on the chemical equilibrium theory. As the authors indicate, encounters and collaboration with people of different specialties are when research results proceed to the next stage.

Research paper: Development and commercialization of laser inspection system to detect surface flaws of machined holes
Saburo OKADA *et al.*
This paper discusses the development and commercialization of an automated, high-precision device system for detecting defects on glossy or mirror surfaces that tend to be missed in visual inspections of inner wall surfaces of machined holes with various diameters in industrial products. The points of each development stage are clearly stated, and the scenario for successful product realization is described such as conducting product development that matches the demands of companies in order to overcome crises such as re-organization of the research institute and the Lehman Shock.

Commentary: Contributing to the SpaceWire international standard
—Successful factors for the development of a de jure standard—
Hiroki HIHARA *et al.*
The scenario in which Japanese proposals were adopted is described from the aspects of the technological background and roles of personnel, in the international standardization of SpaceWire that is the communication standard for satellite networks. Applying the Yoshikawa model to the process of development-type standardization work, behavior patterns of the people involved in standardization of Europe, USA, and Japan are analyzed. This may be applied to general international standardization, not limited to the satellite communication standard, and will be useful to people of other fields.

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Challenge towards synthesis of non-silica-based hybrid mesoporous materials

—Level of compositional design and control of mesoporous materials achieved so far—

Tatsuo Kimura

[Translation from *Synthesiology*, Vol.11, No.3, p.115–127 (2018)]

Amphiphilic organic molecules have often been transformed into liquid-crystal structures in their concentrated solutions. This paper focuses on a group of porous materials, called "ordered mesoporous materials." Ordered mesoporous materials have nanostructures that replicate liquid-crystal structures. I report on the current level of compositional design that can be realized using mesoporous materials. In addition to silica-based materials, various inorganic compositions have been recently considered as possible alternatives. I have been striving to develop a more difficult method to obtain hybrid mesoporous materials in a non-silica-based system. To realize this, I have selected novel chemical resources for the synthesis of ordered mesoporous materials, proposed a new synthetic route, and realized reactivity control of such chemical resources and their functional design.

Keywords : Mesoporous structure, supramolecular template, compositional design, non-silica-based material, inorganic-organic hybrid framework

1 Introduction

Porous materials are materials that possess a large amount of void space within. Therefore, material surfaces are exposed in large amounts. This means that application is expected to various extended uses that utilize the characteristic of having an extremely large amount of surface area. The materials are categorized into microporous, mesoporous, and macroporous materials according to the pore size. According to the definitions of the International Union of Pure and Applied Chemistry (IUPAC), the microporous, mesoporous, and macroporous materials have pore size distributions in the ranges of 2 nm or less, 2–50 nm, and 50 nm or more, respectively. A representative industrially important microporous material is zeolite (crystalline aluminosilicate) used in oil refinery processes and chemical product syntheses, and ion exchange zeolite is used as a catalyst for cleansing automobile exhaust gases. Silica gel used as a desiccant is the most famous mesoporous material, and this is also used as a filler in columns of analysis devices as an adsorption-separation agent.

Among the mesoporous materials, one must go back to around 1990 for the discovery of a silica porous body that was synthesized using a self-assembling characteristic of a surfactant (amphiphilic organic molecules).[1] It drew worldwide interest due to the expectation for development of new uses for the structural characteristic unseen before in which uniform mesopores were arranged orderly in an extremely narrow pore size distribution.[2] Although the image is a common sight now, one can imagine that the transmission electron microscope (TEM) photograph that showed the uniform ordered mesopores that was published in *Nature* at the time of its discovery made a great impression on materials researchers.[3] Figure 1 (top) shows a typical analysis result of mesoporous silica. A nanoscale ordered structure can be seen from X-ray diffraction measurement in the low-angle range, and it is possible to directly visualize the structure through TEM observation. Also, by analyzing the shape of nitrogen adsorption isotherms, it is possible to calculate a specific surface area, pore capacity, and pore size distribution. However, impurities or materials that have not turned porous may be mixed. Therefore, to determine the success of mesoporous silica synthesis, it is necessary to see not only the uniformity and periodicity of mesopores, but also various analysis results comprehensively.

Figure 1 (bottom) is a summary of the examples of application development including the possibility of achieving mesoporosity through various material compositions. Normally, application development using functions of oxides is conducted. In cases of porous materials, development of applications as catalyst carriers and adsorbents is conducted to make use of pore space. In order to add functions that are not expressed with silica alone, functions (acidity, oxidation function, etc.) are added by introducing heteroelements, or organic groups are incorporated into the silica framework as in mesoporous organosilica (hybrid mesoporous material

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in which a silica framework, an inorganic species, and organic groups are compounded at molecular scale). In any case, the catalyst functions are designed by introducing components necessary for the achievement of functional expression. Since there is no other option than to use the pore environment provided by the final product, the design of the pore environment is important for the expressed function to efficiently progress. In this research, I present the difficulty of synthesis research and the level that I have currently achieved, taking as examples the mesoporous material composition design and the synthesis method of non-silica-based hybrid mesoporous materials for which control technologies have been advanced. Ultimately, if I utilize elemental technologies needed for functional design that were clarified in silica, it will be possible to design a chemical reaction field at nanoscale where an expressed function can progress efficiently as possible.

Figure 2 shows the types of surfactants generally used for the synthesis of mesoporous silica, the range of pore size control (1–100 nm or more), and comparison with a typical molecular size. In microporous materials, only small molecules such as benzene can be utilized. On the other hand, mesoporous materials can take in larger substrates into the pores. For example, there is rising expectation that the pores may be used as nanoscale synthesis vessels for fine chemicals or solidification media of huge functional molecules such as protein and enzymes (box of red dotted line). There are also possibilities of increasing performance that arise from increased molecular diffusion. As a guideline, the pore size range at which such functions are expressed effectively is shown (box of blue dotted line). Research of mesoporous materials progressed into a global research field due to the uniqueness of the material. Moreover, there was increased expectation that mesoporous materials might be synthesized by methods similar to silica but with non-silica compositions.[4]

If mesoporous organosilica precedingly developed from silica materials was from research activities “specializing on functional design,” the primary research strategy of this research is “to design the pore environment.” In general, hydroxyl groups are present on a silica surface, and it is said that some degree of hydrophilic property is seen on a flat surface. However, it is reported that hydrophobic behavior is seen in concave surfaces inside mesopores. Since mesoporous organosilica has hydrophobic organic groups within its framework exposed on mesopore surfaces, only application development assuming a hydrophobic pore environment can be conducted. Against this background, I set the initial goal of this research as “designing a hydrophilic surface structure” that was never observed in other mesoporous materials. Mesoporous aluminophosphate has extremely low structural stability and was not suitable for application development. However, it was reported to be the only mesoporous material with a hydrophilic surface structure,[5] and I decided to propose a design guideline for a surface structure using this finding as a hint.

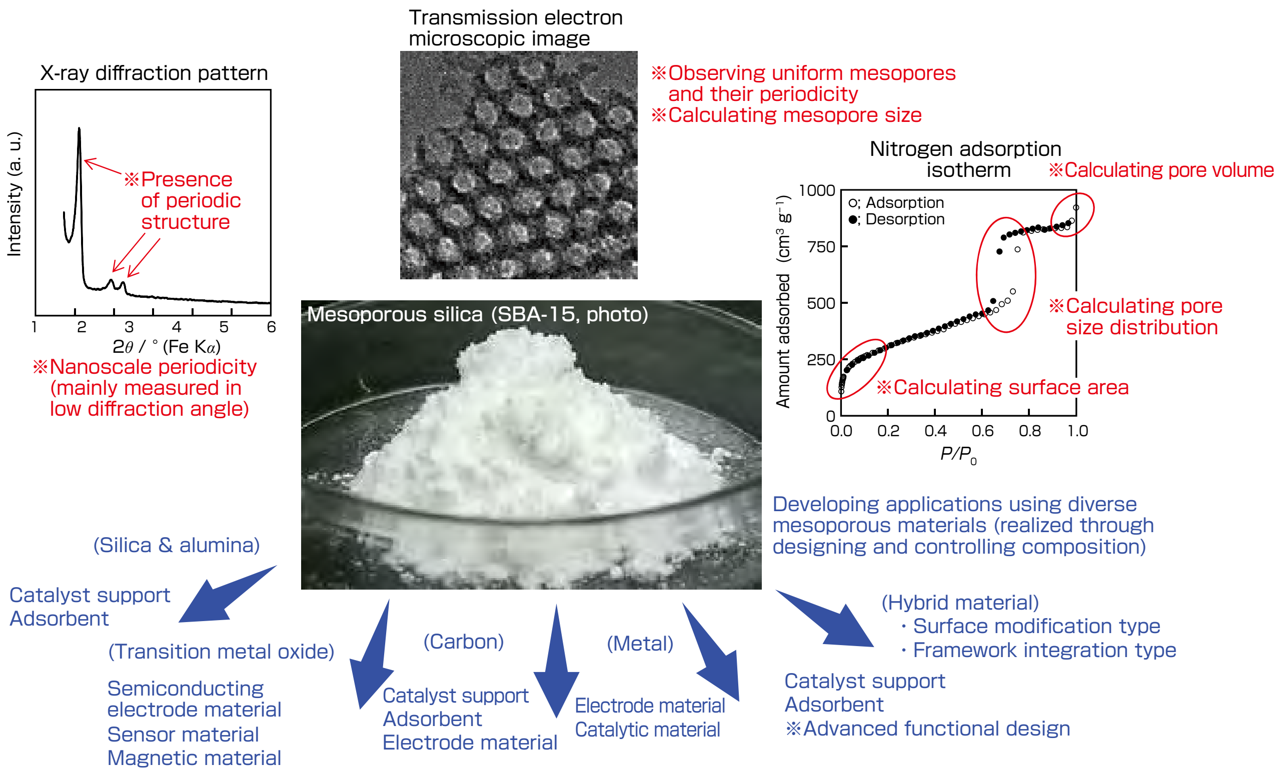

Fig. 1 Representative analyses of mesoporous silica and examples of developing applications using diverse mesoporous materials

2 Summary of extension of compositional control range for mesoporous materials and technological issues

In this study, I discuss raising the level of control technology for framework composition of mesoporous materials. From the viewpoint of the synthesis chemistry, I summarize the difficulty of achieving mesoporosity in non-silica-based materials, the technological issues that prevent inorganic-organic compounding, while comparing with hybridization (inorganic-organic compounding) of the silica materials. The main elemental technologies of synthetic research for mesoporous materials are as follows:

(1) Selection of appropriate inorganic starting material, and reaction control of inorganic species in solution,
(2) Design of interaction between inorganic species and amphiphilic organic molecules in solution,
(3) Understanding the self-assembly behaviors of amphiphilic molecules that are newly produced by interaction,
(4) Adjustment of the process of liquid-crystal-like structure formation and the following polymerization of inorganic species,
(5) Development of removal method of amphiphilic organic molecules, and
(6) Process design assuming application development in thin films, powders, and others.

Figure 3 shows these elemental technologies in conjunction with each stage of formation of mesoporous silica precursors. The most important point here is to understand all elemental technologies comprehensively. That is, this research result, or the composition design technology of advanced mesoporous materials, could not have been made unless these elemental technologies were integrated.

The formation mechanism of mesoporous silica precursors that was shown immediately after the discovery of mesoporous silica drew controversy. If one understood that the precursors were formed by the liquid crystal template route as shown in Fig. 3 (top), it was sufficient to believe that space design at nanometer level could be done easily inside oxide materials. However, as a result of follow-up investigation, it was found that in most cases, the precursors (composite of liquid-crystal-like structured silica and amphiphilic organic molecules) are produced by concerted organization, as shown in Fig. 3 (bottom), rather than the liquid crystal template route. First, the hydrophilic regions of amphiphilic organic molecules interact with the dissolved silicate species in oligomer form. At this point, if the molecular size of inorganic species is too big, precursors with structural order cannot be obtained, or precipitates are formed beforehand. If interaction with inorganic species goes well, it can be considered that new inorganic-organic composite molecules with amphiphilic properties have been formed. If self-assembly of this composite molecule and the bonding of inorganic species occur simultaneously (concertedly), precursors with high structural order containing liquid crystal structures are obtained. Finally, when organic molecules are removed by firing or other methods so the structural order is not broken, an orderly mesospace is formed inside the material.

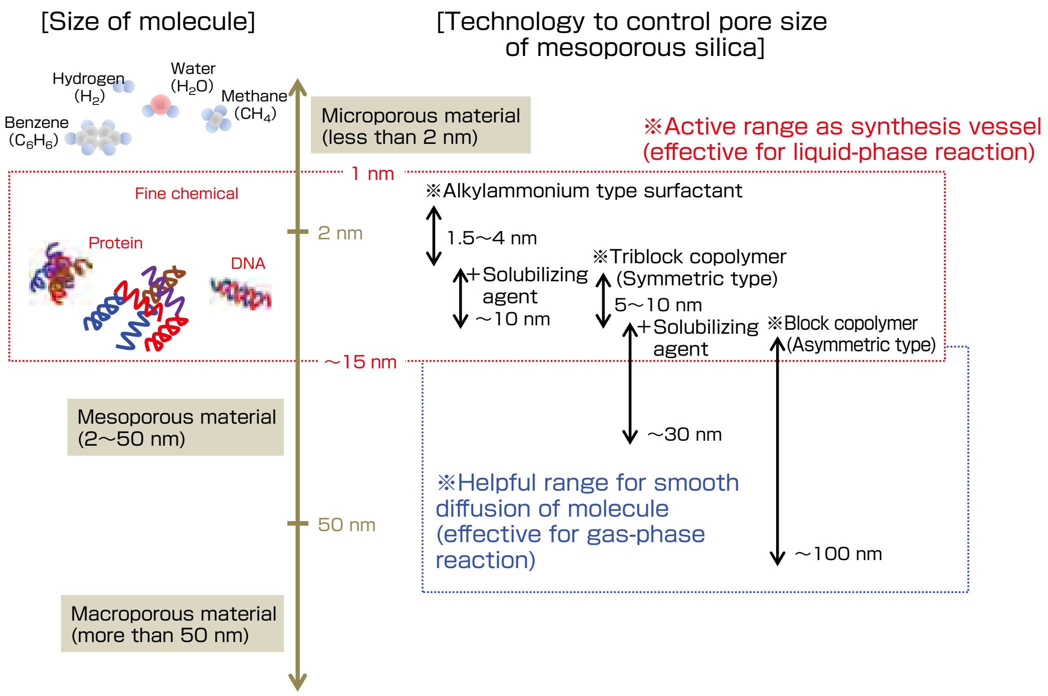

Fig. 2 Comparison between molecule size and range of pore size control of mesoporous silica

2.1 From silica to its hybrid (organosilica)

For cases in which synthesis is started from silica starting materials such as tetra-alkoxysilane, there are sufficient findings in silicate chemistry.[6] This means that the synthesis is done in an environment in which hydrolysis and polycondensation reactions can be controlled easily in solution. Moreover, because basic scientific understanding that conditions and reaction processes of dissolved silicate species can be traced by ^{29}Si NMR has grown, it is actually possible to synthesize diverse types of mesoporous silica. Inorganic-organic compounding of the framework in silica materials can be relatively easily achieved, even by using silane compounds bridged with organic groups as starter materials.[7] Although reactivity of silane compounds may change, findings of silicate chemistry can be used fully, such as synthesizing by selecting either acidic or alkaline conditions. As shown in Fig. 4, there are reports of mesoporous organosilica synthesis from silane compounds bridged by various organic groups. Also, inorganic and organic groups are arranged alternately at molecular scale, and intermolecular interaction between organic groups within silane compounds may give periodicity to the arrangement of the organic and inorganic species.[8][9] Simple organic groups are only expected to have the role of merely making pore surfaces hydrophobic. On the other hand, there is work done on utilizing the functionality or designability of organic groups themselves. For example, technology to capture light energy is being studied by forming a metal complex using bipyridine arranged as footholds on framework surfaces.[10]

2.2 From silica to non-silica: Difficulty to synthesize non-silica-based mesoporous materials

The knowledge of silicate chemistry does not apply at all to the synthesis of non-silica-based mesoporous materials. Here, I shall explain using oxides such as alumina and titania as representatives of non-silica-based materials. Non-silica-based inorganic starters have severe reactions in solution. Therefore, chemical modifiers are used or non-aqueous systems are applied for reactions, and various other measures are reported as control methods to delay the general sol-gel reaction. However, combining such reaction control technology to the concerted organization route (Fig. 3) to obtain precursors for mesoporous materials has not been done fully. Since efforts are only mainly applied to the initial stage of the reaction, subsequent bond formation among inorganic species cannot be controlled. Therefore, self-assembly and framework formation are not adjusted to an appropriate speed, and inorganic materials are precipitated without being fully incorporated into amphiphilic organic molecules. Such behavior can be understood as follows: strength of bond energy (covalent bond) of inorganic framework >> energy of interaction (electrostatic interaction, hydrogen bond, etc.) of inorganic species and amphiphilic organic molecule > power of energy of self-assembly. To obtain a mesoporous structure with high structural order, how to control bond formation of inorganic species in solution is the most important component.

For non-silica-based materials for which reactions are difficult to control, precision is required to control the reaction in solution. In other words, the fact that most reports for alumina and titania are limited to films shows

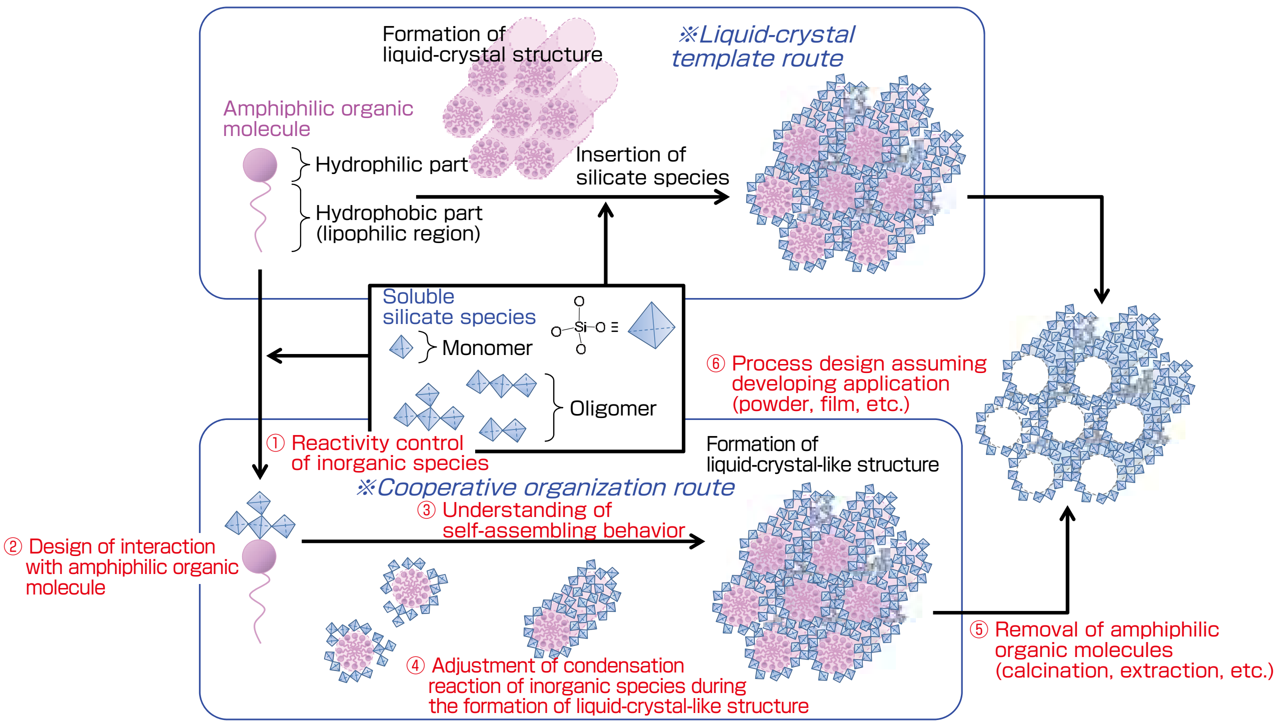

Fig. 3 Formation mechanism of mesoporous silica: Liquid-crystal templating and cooperative organization routes

the difficulty of obtaining mesoporous material from non-silica-based materials. To prevent formation of precipitation while controlling bond formation of inorganic species, first, a transparent precursor solution is prepared. Next, a solvent evaporation method[11][12] is used to rapidly form a mesoporous structure to obtain a highly ordered mesoporous film. Preparation of a precursor solution is done mainly with an ethanol solvent. However, an alkoxide starting material cannot interact with the hydrophobic region of amphiphilic organic molecules. To slowly promote progression of hydrolysis and condensation reactions of alkoxide materials, a small amount of hydrochloric acid is added. Even then, when amphiphilic organic molecules and dissolved inorganic species interact, solvent evaporation is immediately promoted by processes such as spin coat and spray-dry, to bring them to a quick finish as precursors of mesoporous materials.[13] Even more troublesome is the fact that polymerization reaction of oxide materials progresses even after film formation. As a desperate measure to forcibly delay this reaction, sometimes, the product may be placed in a freezer (about -20 °C).

In the actual follow-up test, it has been confirmed that the structural order greatly decreases if it is not placed in a freezer. This is fine for goal-oriented basic research of performance evaluation of materials or for studying structure correlation. However, this cannot lead to process design for mass synthesis or mass production for practical use. Moreover, surprisingly, a majority of non-silica-based mesoporous materials presented as powder samples in published papers are actually synthesized by a solvent evaporation method. I have heard that there are cases in which the precursor solution is spread thinly as possible and then samples are collected by film formation. As a result of the difficulty of synthesizing non-silica-based mesoporous materials, such heavy-handed synthesis methods are used worldwide. Synthesis research mindful of process design suitable for practical application cannot be done unless understanding is deepened for the reactivity of inorganic materials and composition control technology is advanced in a true sense. In addition, there are expectations for the material to be used as a catalyst carrier by only using the effect of increased surface area like alumina. In most cases, sufficient function expression cannot be expected unless the crystalline property of the oxide framework is increased. For those that realized the synthesis of mesoporous film using oxides of transition metal, proposals were made for new application technologies as device parts such as sensor materials or electrode materials. Therefore, I did see effects of increasing adsorption volume of photo-responsive molecules or adsorption sites of sensing target components. However, since it was not possible to increase the crystalline property sufficiently while maintaining porosity derived from the mesoporous structure, it seemed that the effect of achieving mesoporosity was limited.

3 Non-silica-based hybrid mesoporous material: Toward building of substance group

As discussed above, functional design derived from silica cannot be expected. In starting this research, it was necessary to simultaneously realize inorganic-organic compounding of the framework, in addition to achieving mesoporosity in non-silica-based materials. I set opening the way to this advanced material design as the ultimate goal of synthesis study. When I started, in reality, there was not even a way of synthesizing non-silica-based hybrid mesoporous materials. If I could develop a universal synthesis technology for a mesoporous material that was non-silica-based and was composed of an inorganic-organic framework, I could clear the way toward advanced material design, such as arbitrarily changing the environment inside the nanospace from hydrophobic to hydrophilic using properties of non-silica material surfaces,

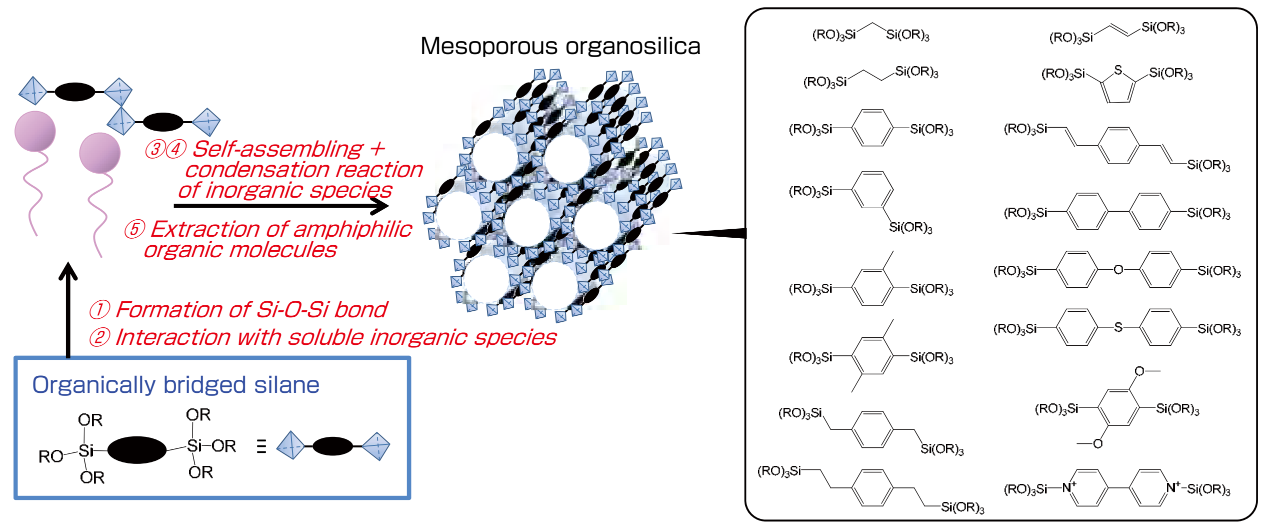

Fig. 4 Synthesis of mesoporous organosilica: Variation of organic groups (with the number of significant technologies shown in Fig. 3)

or compounding with functions derived from inorganic species. As mentioned earlier, the progress from silica to organosilica went relatively smoothly. On the other hand, why did not the research for non-silica oxides lead to synthesis research for inorganic-organic compounding? It was probably because rather than the difficulty in controlling the reaction of a starting material, there was no such starting material. Other than silica, alkoxide materials or similar compounds that include bridged organic groups in the structure are not available commercially. In reality their synthesis methods are not reported. I found three types of tin compounds that were bridged by organic groups.[14] If I had to develop synthesis methods of such compounds for each metal species, I would have to spend an enormous amount of energy on preparing raw materials for mesoporous materials, and I thought this was not a realistic approach.

3.1 Proposal of synthesis method: Limit of organically bridged phosphonic acid

From the above background, I have searched for compounds that are capable of bond formation with various metal species and included bridged organic groups within the molecular structure. As a result, I proposed the use of bisphosphonate compounds (similar to phosphoric acid that are bridged with organic groups). The synthesis technology for phosphonic acid was almost established. Moreover, in the synthesis of metal phosphates, for example, if the mol of phosphoric acid in solution was the same as the metal species, the reaction of the metal species would be inhibited, and I thought this advantage could be utilized. The mesoporous material was synthesized using commercially available phosphonates bridged with organic groups. Since I set the design of a “hydrophilic surface structure” as the first research goal, initially I spent my efforts on the synthesis of mesoporous aluminum phosphonates in which aluminum was the metal species. As a result, I was able to propose a synthesis route of a non-silica-based hybrid mesoporous material, as shown in Fig. 5 for the first time.[15] For verification, I selected the simplest reaction of methylene bridged phosphonates and aluminium isopropoxide that is the aluminum source, and conducted the synthesis using an alkyltrimethylammonium (C_nTMA) surfactant under alkaline conditions. The facts that the structural order could not be obtained sufficiently when achieving mesoporosity and that low-temperature firing had to be used since the surfactant could not be extracted (the bond between phosphorus atoms and bridged organic groups is partially disconnected) were seen as new issues.

The first issue, to improve the orderliness of the mesoporous structure, was not too difficult.[16] The structural order improved greatly just by using methylene bridged phosphonates and aluminum chloride ($AlCl_3$) with suitable reactivity as the aluminum source, and by changing the synthetic condition to an acid condition. As a surfactant,

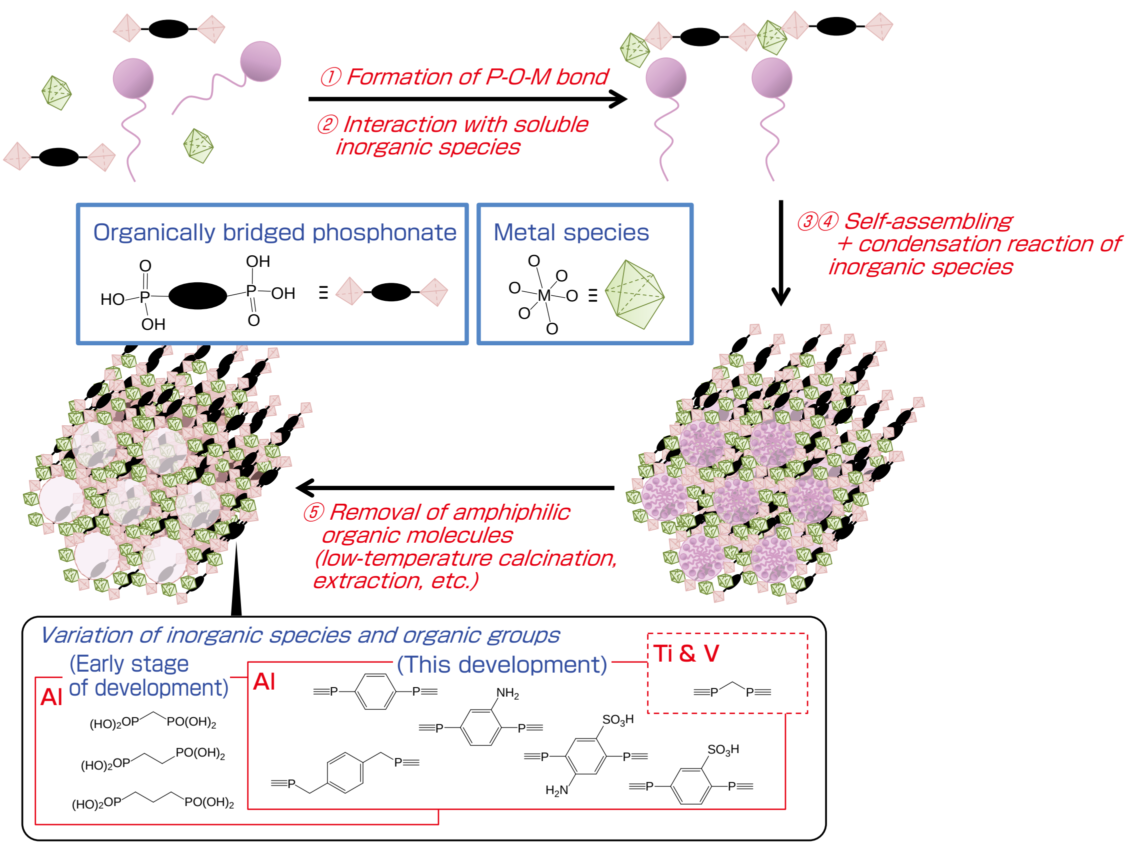

Fig. 5 Proposal of the synthesis route from organically bridged phosphonate compound, and progress in composition control technology (with the number of significant technologies shown in Fig. 3)

not only C_nTMA, but also alkyl polyoxyether (C_nEO_m) or polyoxyethylene-polyoxypropylene-polyoxyethylene triblock copolymer ($EO_nPO_mEO_n$) could be used.[16][17] By using the changes in alkyl chain length and different polymerization number, it was confirmed that the pore size could be controlled in the range from around 2 nm to little less than 10 nm. At this stage, the surfactant was removed by low-temperature (for example 400 °C) firing, and therefore, I could only make products with bridged organic groups with relatively high heat resistance, such as methylene groups or ethylene groups. Using a similar synthesis method, for example, I attempted synthesis from benzene bridged phosphonates that could be functionally designed and had high heat resistance. However, I was unable to obtain mesoporous aluminum phosphonates with high structural order. The details will be explained later, but this presented the limit of synthesizing from organically bridged phosphonates. That is, I reached the conclusion that it was difficult to extend this substance unless there was a breakthrough in the precision reactivity control of the starting material.

To solve the second issue, the development of an extraction method of the surfactant, efforts were made by trial and error. As a result, it was found that C_nEO_m and $EO_nPO_mEO_n$ could be dissolved (removed) simply by heating in an acetone solvent.[18][19] I achieved a situation in which a hybrid framework could be designed without considering heat resistance of bridged organic groups, and this was an extremely important result that pushed the research forward. It is thought that the phosphate (P-OH) group on the solid surface showing acidity acted as a catalyst to break down the EO and PO units. In mesoporous aluminophosphate, the stability of the mesoporous structure was so low that the structural order gradually degenerated even when there was steam present. Therefore, in a case of aluminum phosphonates that contained an aluminophosphate-like framework, it was important to do acetone treatment so there would be few water (H_2O) molecules as possible. It was thought impossible to conduct performance evaluation for mesospace with hydrophilic environment unless this low stability was resolved. Fortunately, it was found that the stability of a mesoporous structure increased with the introduction of bridged organic groups. As a hydrophobic organic group was introduced proximal to the aluminophosphate-like framework, hydrolysis by H_2O molecules was inhibited to some degree. As a result of introducing an organic group, the material surface became somewhat hydrophobic. Understanding this as being a local structure, the aluminophosphate-like structure that is the point of absorption of H_2O molecules will remain exposed at the pore surface. That is, the road to performance evaluation of a hydrophilic surface environment or its proximity was not closed.

3.2 Reactivity control of starting material: Possibility of phosphonate compounds

The reactivity of bisphosphonate is changed dramatically when organic groups with high electron density such as benzene linkers are bridged. In this case, the reactivity with $AlCl_3$ was decreased, and it was thought that mesoporous materials could not be synthesized.[20] Here, synthesis was done using a mixed solvent of ethanol-water, but it was necessary to consider the solubility of organically bridged phosphonates in the solvent. For example, xylene bridged phosphonate was not dissolved in an ethanol-water mixed solvent, and even preparation of a precursor solution could not be done. In synthesis from bisphosphonates bridged by simple alkyl groups, mesoporous materials could be synthesized in an optimal synthetic condition even if organic groups were slightly different. When the property changed greatly as in benzene linker, the optimal synthetic condition could no longer be applied. For example, in the synthesis of mesoporous metal phosphate, the importance of appropriately selecting the difference between acid and alkaline of the starting material has been reported.[21] In accordance with reactivity in the early reaction, in this synthesis system, when bisphosphonate ester $(H_5C_2O)_2OP\text{-}R\text{-}PO(OC_2H_5)_2$ was used as a starting material instead of its acid form $(HO)_2OP\text{-}R\text{-}PO(OH)_2$ (R = bridged organic group), its reactivity with $AlCl_3$ was too high and a gel was formed. Considering that the reactivity of phosphonates was insufficient, it was noted that reactivity with $AlCl_3$ could be designed appropriately if there was a similar compound with reactivity somewhere in between that of phosphonates and esters.

3.3 Diversification of organic groups: From alkyl group to aromatic compounds

Though it is referred to as a phosphonate compound here, phosphonic acid is made by treating phosphonate esters in excessive amount of aqueous hydrochloric acid solution. If the treatment is done in conditions in which hydrochloric acid is lacking against the number of ester groups, a compound in an intermediate condition, that is, a phosphonate compound in which acid and esters coexist in the same molecular structure is obtained. I thought it would be possible to conduct continuous reaction control if the percentage was changed arbitrary. Here, the actual synthesis of mesoporous aluminum phosphonates from benzene bridged phosphonate esters when $AlCl_3$ was used as the aluminum source will be explained. Synthesis was done using as starting material phosphonate compounds with different degrees of hydrochloride treatment on bisphosphonate esters. As a result I succeeded in obtaining a mesoporous film with extremely high structural order.[22] The result of TEM observation is shown in Fig. 6. It can be seen that the mesopores are arranged evenly throughout the film.

I also obtained a mesoporous film using a similar approach from phosphonate compounds bridged by aromatic compounds such as xylene linkers. Moreover, I worked on increasing my skills to synthesize phosphonate esters in which the molecular structure was arbitrarily designed. Through such efforts, I achieved synthesis of mesoporous films from organically bridged phosphonate compounds in which amino groups (-NH_2) or sulfonic acid groups (-SO_3H) were added to the bridged benzene ring. Figure 5 is a summary of compounds that were introduced as bridged organic groups. Not only simple organic groups, but also various aromatic compounds that could be functionally designed were introduced. This indicates that composition design technology has advanced to the level in which application research could be conducted as in silica. Therefore, the design of a "hydrophilic surface structure" and "survey of its specificity" for the "design of the pore environment" that are primary research strategies of this research have been mostly realized.

3.4 Diversification of inorganic composition: From aluminum to transition metals

Up to this point, the synthesis research that centered on aluminum as metal species was described. As the next step, I worked on the synthesis with metal species other than aluminum and started preliminary experiment for "controlling the pore environment." If diversification of inorganic species could be realized, it would be possible to use properties of inorganic unit surfaces that is traceable to the types of inorganic species. For example, in aluminum, since H_2O molecules can be coordinated until tetracoordinate aluminum species (AlO_4) become 6-coordinate species (AlO_6) as a property of an aluminophosphonate-like inorganic unit, the surface behaves in a hydrophilic manner. Therefore, if synthesis could be done by other metal species, changes will be seen in the H_2O adsorption behavior since those metal species would not become tetracoordinates.

Here, as the simplest example, the result is shown for synthesizing a mesoporous film of metal phosphonates from phosphonate compounds in which methylene bridged phosphonate esters are partially acid treated. It was confirmed that a mesoporous film could be obtained when titanium chloride ($TiCl_4$) was used as the inorganic material, as well as vanadium chloride (VCl_3) that does not have high reactivity. However, since $TiCl_4$ has extremely high reactivity, the precursor solution was prepared using non-aqueous solution (ethanol). It is thought difficult to form a framework (formation of M-O-P bond) from metal species (M) that is present as ion species in solution. If the metal species (M) is capable of covalent bonding through phosphorus (P) and oxygen (O) atoms, I believe a mesoporous material with a new composition can be designed by understanding and controlling the reactivity of those metal species in solution.

4 Process design assuming practical application using mesoporous material

In this paper, the major elemental technologies for synthesis research of mesoporous materials are categorized from (1) to (6). For example, as shown in Fig. 5, it was explained that I was able to synthesize a hybrid mesoporous material using non-silica as a result of integrating (1) to (5). I present my conclusion by providing a discussion of the final elemental technology "(6) Process design assuming application development." Normally, materials development starts from fulfilling the demand for high performance against "social demand (application development)" as shown in Fig. 7 (top). If it is a replacement of an existing material (improvement research), the usage is already predetermined. Therefore, if high performance can be achieved through a similar synthesis system, the level of performance improvement that can be achieved is confirmed. If there are no issues about the cost of raw materials or manufacturing processes, it

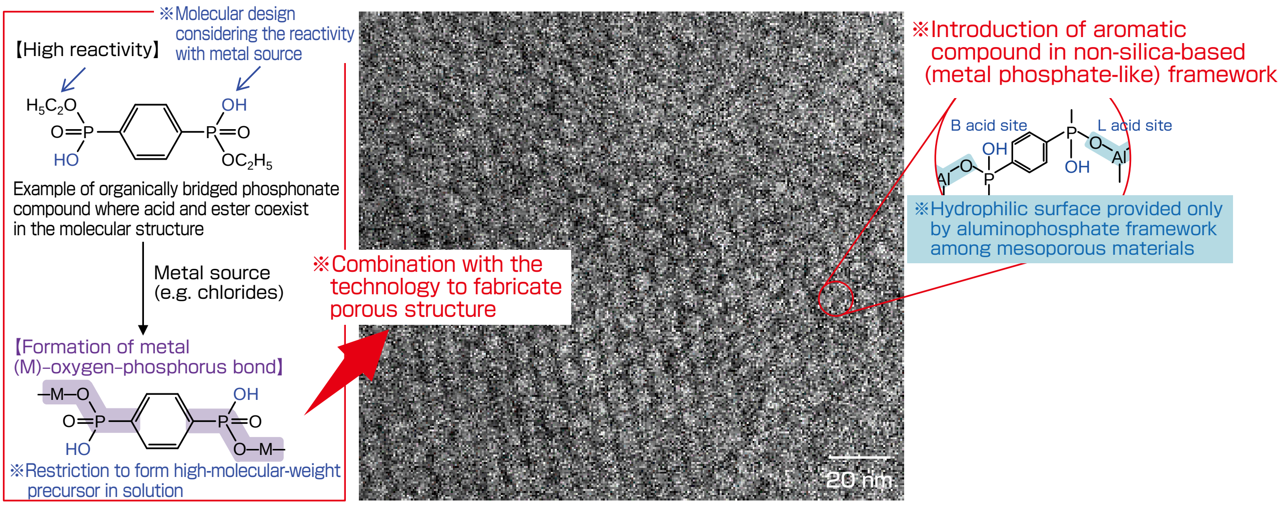

Fig. 6 Reaction of benzene bridged phosphonate compound and metal source, which combined with the technology to fabricate mesoporous structure

will become a candidate alternative material. On the other hand, in cases that start with a discovery of a new material (dramatically enhanced function), there is a possibility of technological innovation. However, I all know that there are plenty of technological hurdles that must be overcome before practical application. Depending on how the new material is to be used, even the synthesis method that was first found may have to be altered. In general, this corresponds to the development of control technology for "composition," "structure," and "form" in synthesis research.

In a case of materials development in which mesoporous technology is used, materials synthesis is attempted by clarifying the developmental factors that are to be targeted. This is explained in Fig. 7 (bottom). If the performance in demand is clear, some hints may be obtained for candidate composition to achieve that function through surveys of existing research. Limited to the cases in which performance improvement can be expected through structure control (in this research, mainly achieving mesoporosity), I start the development of porosity technology using materials with candidate composition. If the form in which it will be used is set, the process of sample preparation as a film or powder can be determined. For example, say that one prepares a homogenous precursor solution, particularly a transparent one. In this case, separation of films and powder is possible only by coating or spray-drying, as the process used for both is solvent evaporation by which precursors for mesoporous materials is made. Therefore, development of structural control and form control can be construed as development of elemental technologies that can be conducted together. In the sense that formation of mesoposority is conducted for selected candidate composition, the composition is not the target of control. In synthesis research for mesoporous materials like this research, it is most important to properly understand the reactivity of raw materials in solution to advance mesoporosity technology.

There is another important point in achieving assumed performances to the maximum. In most cases, the precursor materials of mesoporous materials are obtained with inorganic frameworks of amorphous conditions, and it is necessary to crystallize the inorganic frameworks after achieving mesoporosity, if the expected performance is derived from the functional expression of crystalline materials. Since the inorganic framework can be crystallized by heat treatment (firing), additional process is not necessary. As mentioned before, if crystallization goes too far, the mesoporous structure disintegrates in most cases. Therefore, maximizing the crystallized ingredient without losing the effect of mesoporosity will be the aim of material design. Also, ones with larger pore sizes tend to have mesoporous structures that do not disintegrate even with crystallization. In such cases, the merit of "improved dispersibility" is obtained as well as "increased crystallization." Therefore, it is important to develop a precise synthesis technology by which the performance is maximized through the effects of surface area, crystallization, and dispersibility.

5 Future issues and prospects

The synthesis technology for "non-silica-based hybrid mesoporous materials" that was developed in this research showed major progress and has potential for greatly expanding the compositional control range of ordered mesoporous materials. However, mesoporous aluminum phosphonates, which were synthesized using phosphonic acid bridged with an alkyl group at the start of the research, was collected as powder.[15]-[19] On the other hand, formation of films was the main theme of research for phosphonate compounds bridged with various aromatic compounds that were recently successfully developed.[22] Fortunately, powder

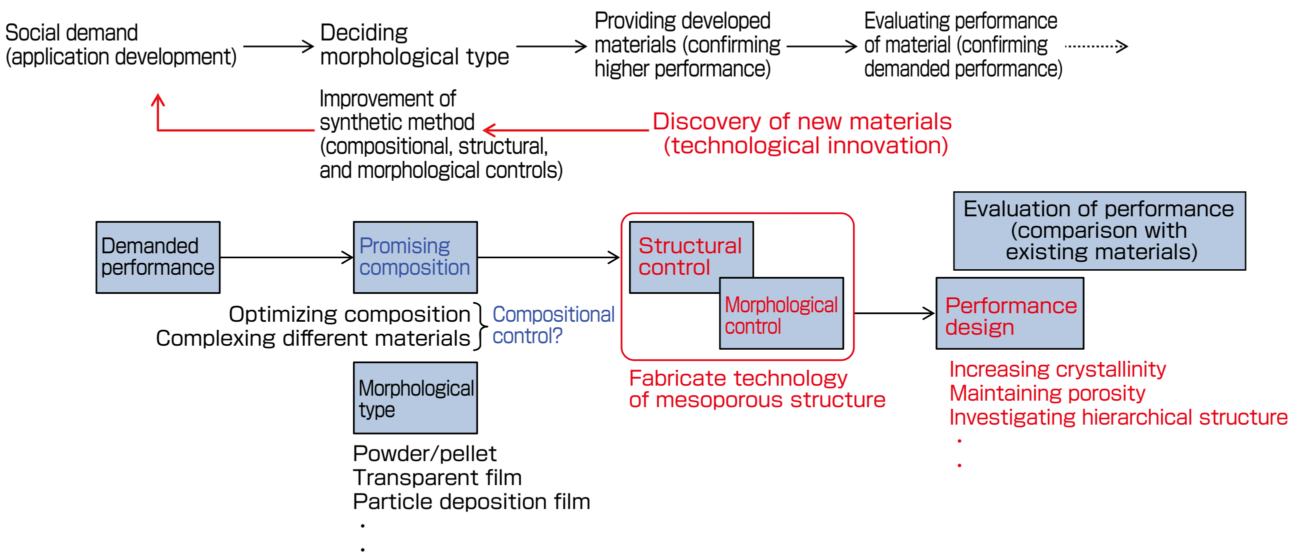

Fig. 7 Basic guideline for the materials design to develop applications

synthesis of mesoporous aluminum phosphonates was researched using a spray-drying process that is known as a synthesis method including a solvent evaporation process similar to film synthesis,[23][24] and the understanding of this process was advanced.[25] Therefore, according to the demand of application development, it will become possible to provide various mesoporous metal phosphonates that were designed recently or will be designed in the future, as powder samples.

The "reactivity control of phosphonate compounds" was a breakthrough to greatly advance this research. One of the future prospects that emerged from the study is the expansion of the types of organic groups in the framework. Not limited to the organic groups seen in commercial phosphonate compounds, it is necessary to design molecular structures of bridged organic groups assuming all sorts of chemical approaches, including organic synthesis of phosphonate esters with desired bridged organic groups as well as addition of functional groups to phosphonate esters. I believe functional design using organic groups in the framework will be done with future orientation. In this research approach, reactivity can be controlled continuously through partial acid treatment of phosphonate esters. However, I do not think this is a universal method for all inorganic materials. Moreover, I have not reached the level of synthesis considering crystallinity of inorganic frameworks of mesoporous materials, and new ideas are necessary for its realization.

It is not certain that the concept of continuous reactivity control can be introduced to the side of inorganic species. In the future, it is necessary to develop synthesis methods for diverse mesoporous materials, being mindful not only of understanding the reactivity of inorganic materials during the early stages of reaction but also of how to control the reactivity of inorganic species that is taking place continuously in solution. Moreover, I hope to advance to a synthesis method that allows maximization of the crystalline property of inorganic frameworks. By doing so, the diversification of inorganic species to this point can be positioned as efforts toward "designing the pore environment." "Functional design derived from inorganic species" can be added to future design guidelines. Simple usages of mesoporous metal phosphonates are summarized in several papers.[26]-[29] In the sense of future-oriented material design, metal phosphate and its metal oxides are predicted to show similar properties that derive from the property of metal species. Therefore, in the future, I wish to discuss possibilities for materials that act as alternatives in application development for non-silica-based oxide materials of which achievement of mesoporosity and powder synthesis technologies are currently not sufficiently established.

Acknowledgement

In conducting this research, I had pride of a synthesis researcher and the will to succeed despite insufficient funding. Therefore, I made sure my papers were published in illustrious international academic journals, and continued to generate steady research results, sometimes through collaboration with exterior research institutions. My efforts have come to fruition. Very recently, I was able to greatly advance this research, through the support of the Grant-in-Aid for Scientific Research (B), "Development of a precise synthesis technology for non-silica-based hybrid mesoporous materials by the molecular structure design (FY 2014–2016)" Japan Society for the Promotion of Science (JSPS KAKENHI Grant No. 26288110). I am grateful for this opportunity.

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Discussions with Reviewers

1 Overall

Comment (Toshimi Shimizu and Motoyuki Akamatsu, AIST)

This paper describes, in scenario style, the details of the course by which synthesis of a non-silica-based hybrid mesoporous material, which was considered difficult, was achieved. The challenging strategies and tactics involve selection of new starting materials and utilization of unconventional synthesis routes. I think it is significant that you were able to extend the composition and structure of mesoporous materials as a result of this research. The paper demonstrates that the integration of cumulative elemental technologies is important, using tools of sub-nanometer scale chemical species such as organic and metal species. Hence, the paper is suitable for publication in *Synthesiology.*

2 Synthesis and integration of elemental technologies

Comment & Question (Toshimi Shimizu)

The author uses Fig. 3 to show individual items (1) to (6) as elemental technologies for the synthesis research for mesoporous materials, and emphasizes that their integration is essential in composition design control of advanced mesoporous materials. However, looking at the subchapter titles of the first draft of this paper, rather than explanations of elemental technologies from (1) to (6), you provide explanations of new chemical materials and synthesis routes from silica to organosilica, alumina and titania, and then non-silica-based hybrid mesoporous materials. In other words, there is no specific explanation about the integration of elemental technologies. To deepen the readers' understanding, I think you should clearly state what correlation exists between newly developed chemical materials and synthesis routes and the elemental technologies (1) to (6), as well as specific contents about the integration of elemental technologies. Also, here is a question: Is the route newly developed by the author (Fig. 4 and Fig. 5) neither the liquid crystal template route shown in Fig. 3 nor the concerted organization route?

Answer (Tatsuo Kimura)

In the process of synthesizing mesoporous silica shown in

Fig. 1, the elemental technologies that must be considered can be categorized into (1) to (6). Excluding elemental technology (6), it is correct in understanding that mesoporous silica is synthesized by their integration. The importance becomes more apparent in materials like non-silica-based materials in which obtaining mesoporosity is difficult. The mesoporous materials cannot be synthesized just by improving individual elemental technologies. Therefore, particularly important technology is how to control bond formation of inorganic species in solution, as stated in elemental technology (1). This is explained in the section about the difficulties of synthesizing non-silica-based mesoporous materials. Moreover, in elemental technology (5) that seems to have low relationship with others, the results are affected by the integrity of the periodic structure (high symmetry) and the degree of condensation of the silicate framework in the mesoporous silica precursor. As a result, it is reported that the mesoporous structure disintegrates during the process of removing amphiphilic organic molecules.

The precursors of mesoporous materials is invariably formed by either the liquid crystal template route or the concerted organization route. In this research a new formation route was not developed. Therefore, I corrected Fig. 4 and Fig. 5 so the details of the synthesis route would correspond to the numbering of elemental technologies. In this research, new composition design technology was proposed and demonstrated using the reaction of organic bridged phosphonate compounds and metal sources to achieve non-silica-based inorganic-organic composites with the ingredients that surround the assembly of amphiphilic organic molecules.

Comment (Motoyuki Akamatsu)

In the first two paragraphs of Chapter 2, you describe the silica-based synthesis route and that synthesis occurs through a concerted organization route. Based on this, you identify six elemental technologies, and your claim is that these six elemental technologies must be integrated for non-silica-based materials. Then you describe the development of non-silica-based mesoporous materials in Chapter 3 in which you state the selection of materials in Subchapter 3.0 and reactivity control in Subchapter 3.2. However, non-specialist readers cannot easily understand which of the six elemental technologies corresponds to which part of the description. Can you make the corresponding relation easier to understand? I think it will make things clear if explanations of the elemental technologies follow the subchapters of Chapter 3.

Answer (Tatsuo Kimura)

I thought I clearly showed the important elemental technologies in the figure for formation mechanism of Fig. 4 (silica-based hybrid materials) and Fig. 5 (non-silica-based hybrid materials). However, it seems that it was difficult to understand for non-specialist readers, so I decided to use the numbering of the six elemental technologies.

3 Research strategy

Comment & Question (Motoyuki Akamatsu)

In Paragraph 5, Chapter 1, you explain that while a study of hybrid mesoporous materials by silica-based materials is R&D that “specializes on functional design,” this research (= non-silica-based hybrid mesoporous materials) took a research strategy (= research policy?) of “designing the pore environment.” What is the essential difference between the two? While silica-based materials followed a goal-oriented approach (of developing a material to realize a certain function), why didn’t you take such a goal-oriented approach here? Please explain your views behind this.

Answer (Tatsuo Kimura)

The essential difference of the research strategies of “specializing in functional design” and “designing the pore environment” is as follows.

Silica-based mesoporous material is made to express functions (acidity, oxidizing function, etc.) that are not expressed with silica alone by introducing different element species into the silicate framework, and at times organic groups are incorporated into the silica framework as in mesoporous organosilica. In any case, design is specific such as for catalyst function or photoresponsivity, and components that are necessary for functional expression (goal oriented) are introduced. However, since there is no option other than using the pore environment provided by the final product, the design of the environment inside the pore is essential for efficient functional expression.

In this research, I discuss the difficulty of synthesis research for hybrid mesoporous materials and the level I have achieved so far. In other words, the discussion is about the methodology for designing the pore environment. Ultimately, if I utilize the findings necessary for functional design that had been clarified for silica-based materials, I believe I can design a nanoscale chemical reaction field where functions can be expressed efficiently.

For the question why I did not take a goal-oriented approach for non-silica-based hybrid mesoporous materials that is the target of this research, that is because I have not reached the stage in which I can freely synthesize with a goal in mind. Even for a silica-based mesoporous material, it cannot be synthesized on demand. Because there are overwhelming number of reports and the selection range has expanded, it merely looks like there are lots of accomplishments in “specializing on functional design” that is actually the next phase of research. In non-silica-based materials, as described in this paper, the reactivity of raw materials is fast, and an enormous amount of experimental samples are needed to synthesize just one type of mesoporous material with a certain inorganic composition. Therefore, I think there is great significance that I opened a path toward diversification of non-silica-based hybrid mesoporous materials through an approach of continuous control of reactivity of the raw materials, as shown in this study.

4 New properties of materials and expanded usage

Comment (Toshimi Shimizu)

By extending the composition of mesoporous materials from the current silica to organosilica, or from silica to alumina and then to titania, and from organosilica to non-silica hybrids, I can expect the expansion of structure, function, and property value of mesoporous materials. To strengthen the significance of this paper, how about adding specific new properties and advantages I can expect through the development of a universal synthesis method for non-silica-based hybrid mesoporous materials? Also, if possible, I think it will help the readers understand the research objective if you add the potential and expectations on how such development will contribute to industrial and social demands. There is a section at the bottom of Fig. 1 that points to this. However, it is organized by items “silica and alumina,” “transition metal oxides,” “carbon,” “metals,” and “hybrid materials,” and they do not correspond to the terminology used in this paper like “non-silica-based” or “non-silica-based hybrid.”

Answer (Tatsuo Kimura)

For the point that you indicated, I added a text to Paragraph 3, Chapter 1 of the final draft. Also, for the section at the bottom of Fig. 1, I added a simple explanation to Paragraph 3, Chapter 1.

Comment & Question (Motoyuki Akamatsu)

In Chapter 1, you write, “the primary research strategy of this research is the design of pore environment,” and after that you write, “I set the goal as the design of hydrophilic surface structure.” Does this mean the specific goal of “designing the pore environment” is the “designing of the hydrophilic surface structure”? Also, I understood that you set such a goal because, with silica-based materials, you can only do certain application development because they are hydrophobic, but you expect that the usage will expand if they can be made hydrophilic. Can you explain to which fields the usage can expand if you can make it hydrophilic compared to the silica usage shown in Fig. 1?

Answer (Tatsuo Kimura)

As you indicate, I started efforts on “designing the hydrophilic surface structure” for which there were hardly any reports, as my specific goal of “designing the pore environment.” Silica-based and all other mesoporous materials have hydrophobic pore environment, and the pore environment becomes even more hydrophobic in application development using organic groups. Therefore, as the first goal, I attempted to create hydrophilic nanospace directly opposite. Therefore, I assumed usages as catalyst carriers and adsorbents that are major uses of silica-based mesoporous materials as shown in Fig. 1. If I can achieve hydrophilic surfaces, I expect, for example, application development as a chemical reaction field to greatly increase reaction efficiency of chemical substances including hydrophilic regions.

5 Reactivity control of inorganic species

Question (Toshimi Shimizu)

Among elemental technologies, if you change from silica-based to non-silica-based, I think it would be very important what strategies and tactics are employed for (1) reactivity control of inorganic species. To control the reactivity of inorganic species, “a Si-O-Si bond is formed by inserting an organic cross-link region between the silica species, or a P-O-M bond is formed using the interaction of phosphonic acid and metal species by inserting the organic cross-link region between phosphonic acids. If the precursor of porous materials can be formed by self-assembly, the key will be how to extract the core parts (by solvent extraction, low temperature firing, dissolution extraction, etc.).” Is my understanding correct?

Answer (Tatsuo Kimura)

For the understanding of reactivity control of inorganic species, I shall add some explanation particularly to the part you indicated. In the final product, a starting material to which an organically bridged part has been inserted to create an inorganic-organic composite framework is used. However, this changes the reactivity of the raw material but does not directly lead to control.

For example, in the case of silica, the initial reactivity of the material changes by whether chlorosilane (Si-Cl) is used or alkoxylsilane (Si-OR) is used. The formation speed of the Si-O-Si bond changes, not only by hydrolysis of initial reaction, but also by the pH at which the precursor solution is prepared. As the reaction progresses, the hydrophilic regions of the amphiphilic organic molecules and dissolved silicate species interact, a silicate framework is formed as further bond formation takes place as self-assembly progresses, and the precursor of mesoporous silica is obtained. If the reaction of silicate species goes too far, the charge density of the silicate framework decreases, the interacting amphiphilic organic molecules fall off, and only mesoporous silica precursors with lowered integrity of periodic structure are obtained.

In non-silica-based oxides with higher reactivity, a precipitate consisting of only inorganic species is formed. Thus obtaining even the precursor of mesoporous materials becomes impossible. However, in the case of achieving mesoporosity of metal phosphates, phosphates are used as the phosphorus source in almost all cases, and the initial reactivity changes by whether chloride (M-Cl) or alkoxides (M-OR) are used as metal sources. In this case, when phosphates are coexisting at a certain ratio, the metal sources and phosphates react first (P-O-M bond forms), and if the reaction is relatively slow, the following reaction will be relatively mild. In a case in which formation of a P-O-M bond is fast, as in the non-silica-base, it is necessary to inhibit the reactivity in solution, and measures must be taken to prepare an appropriate non-aqueous precursor solution.

Considering the above, even in synthesis using phosphonate compounds instead of phosphates, as explained in the paper, the insertion of organically bridged regions must be done at the same time as the functional design (to realize inorganic-organic composites). Since electron density of phosphorus atoms changes according to the type of organic groups, the initial reactivity of the raw material changes. For the control, this research proposes a new method of controlling reactivity using a starting material in which the ester region (P-OR) and hydroxyl groups (P-OH) coexist in the same molecular structure.

6 Final goal

Comment & Question (Motoyuki Akamatsu)

In the first paragraph of Chapter 3, you write “I set the final goal or ‘point of achievement in synthesis research’ as opening the way to advanced material design,” but does this mean you set as your goal the development of universal synthesis technology? In the latter half of this paragraph, you write about the starting material, but is this the starting material that leads to universal synthesis technology? Also, is this the organically bridged phosphonates that you introduce in Subchapter 3.1? In Discussion 2, in relation to my question, the logical progression of this part is unclear, so please clarify.

Answer (Tatsuo Kimura)

As I wrote immediately after the part you indicated. in the research field of mesoporous materials, the significance of synthesis research of non-silica-based hybrid mesoporous material is opening the way to advanced materials design. As a result, if it is proven that the pore environment can be controlled arbitrarily, advanced application development can be started as the next research phase. There isn’t much meaning in doing usage tests to match the developed material. Therefore, I focus on synthesis research, and aim to provide a series of mesoporous materials with various pore environments. For logical progression, I added corrections in the indicated areas.

High-value materials from incineration residues of burnable garbage

—Production of silica with high specific surface area from "molten slag" and direct transformation of silica to basic raw material for silicon chemical industry—

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[Translation from *Synthesiology*, Vol.11, No.3, p.128–136 (2018)]

We developed a technology for producing silica with high specific surface area from molten slag discharged by a garbage disposal plant. The obtained silica has purity and specific surface area comparable to commercially available synthetic silica. Therefore, this silica can be used in various applications, such as in adsorbents, rubber additives, and coating agents. Previous use of silica recycled from molten slag was limited to aggregate for concrete or asphalt. This research result means that silica recycled from molten slag can now be used as a functional material. We also report on "direct synthesis technology of functional chemicals using silica" as the starting point for this research, the research scenario, and future prospects.

Keywords **:** Molten slag, silica, mesoporous silica, silicon

1 Introduction

1.1 Outline of this research

The authors developed technology to manufacture silica with high specific surface area using molten slag that is generated from incineration plants for municipal garbage, in association with Mitsui E&S Engineering, Co., Ltd. (hereinafter, Mitsui E&S; was called Mitsui Shipbuilding & Engineering Co., Ltd. at the time this research was started; the company name was changed in April 2018). Silica with high specific surface area obtained using the jointly developed technology allows molten slag produced in venous industries (municipal garbage incineration plants) to be utilized as functional materials for arterial industries (chemical and materials industries). In this paper, we report the background for this development, the outline of the technology to advance the use of molten slag, and the future prospects.

1.2 Scenario of this research

Figure 1 shows the overall scenario for matching technological potential (the seeds) and industrial demands (the needs) based on social issues that spurred this research, the road to discovery of technology, and the future prospects. The technological development for manufacturing functional materials from molten slag was conducted by the National Institute of Advanced Industrial Science and Technology (hereinafter, AIST) that was tackling the social issue "Achievement of energy savings in silica chemical products manufacturing" in the project of the New Energy and Industrial Technology Development Organization (NEDO). AIST took on the challenges of finding solutions to topics such as "achievement of high efficiency in garbage treatment by local governments (improvement of profitability)" and "further use of distributed energy facilities" as new research topics. When the project is organized into Fig. 1, one realizes that when research results progress to the next stage, collaboration with human resources with different specialties is important, and it can be seen that the key point is to transmit the significance of the research results in a comprehensive manner when building such collaborative relationships and to have the results be recognized widely throughout society in the process of "result dissemination." Since the field of specialties were totally different among the specialists who had the knowledge of plant operation for waste disposal plants at Mitsui E&S, the researchers working on chemical reaction and molecular design at the Interdisciplinary Research Center for Catalytic Chemistry, AIST, and the researchers studying chemical engineering or surface chemistry at the Research Institute for Chemical Process Technology, AIST, there was no opportunity for these people to directly exchange opinions in usual activities such as at academic meetings or at paper presentations. As R&D specialties become more diversified, it is becoming difficult to scan other fields or even science and technology in general. Therefore, events such as the Techno Bridge Fair in which AIST's technological potential is introduced to people of industry are important, so they can get an overview of the research conducted at AIST and to transmit the results widely and systemically. Looking back, it can be said that the breakthrough point of this research was the matchmaking

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between the "seeds" and "needs" of the specialists from totally different fields, and the fact that they were able to start joint research to tackle new topics.

Moreover, in this research, announcement was made in the form of a joint press release for the results of joint research by Mitsui E&S and AIST. As a result, during the first half of the research period, joint research was started to tackle new topics, and in the latter half, we received several inquiries from companies that "wanted to use the silica made from molten slag," and this led to user acquisition for sample distribution work. In the next chapter, details of the activities will be described along the scenario.

2 Background of development

2.1 Current situation of municipal garbage disposal

Ever since the Great East Japan Earthquake, importance is increasing for the usage and popularization of autonomous distributed energy.[1] Municipal garbage disposal plants are capable of utilizing thermal energy generated when disposing garbage, and are drawing attention as distributed energy recovery facilities that are constructed in every local government unit throughout Japan. On the other hand, incineration ashes are produced after burning garbage at these disposal plants, and most of them are buried in landfill sites. However, in some disposal plants, to reduce the volume of ashes, they are melted at high temperature, cooled in water, and recovered as vitrified solids called "molten slag." According to the statistics for FY 2015, there are 1,141 municipal garbage disposal plants in Japan. There are several disposal methods, and there are 220 plants with melting facilities to produce molten slag.[2] Although this is 20 % of the total number of municipal garbage disposal plants, power generation capacity is 3,645 GWh, and this is 45 % of the total 8,175 GWh generated by municipal garbage disposal plants. This is important value of power generation plants.

2.2 Characteristic of molten slag

The main components of molten slag derived from garbage are SiO_2, CaO, and Al_2O_3. Although the composition ratio may fluctuate depending on region or season, the total of these three components is about 70 wt% of the composition, and SiO_2 is about 40–50 %. The production volume of molten slag at municipal garbage disposal plants that treat general waste in Japan reaches about 800,000 ton/year (FY 2015) (Fig. 2). In 2006, two JIS standards (JIS A5031 and JIS A5032; one for concrete aggregate and the other for roads) were established to define the safety of molten slag and to promote its effective use. The items for quality control include physical properties (density, water absorption, abrasion loss, etc.), chemical properties (composition, etc.), and safety tests (elution amount and content of hazardous materials). The current use of molten slag is mostly for construction material. Most uses are for infrastructure construction including about 34 % as road aggregate and about 17 % as concrete aggregate (Fig. 3).[2]

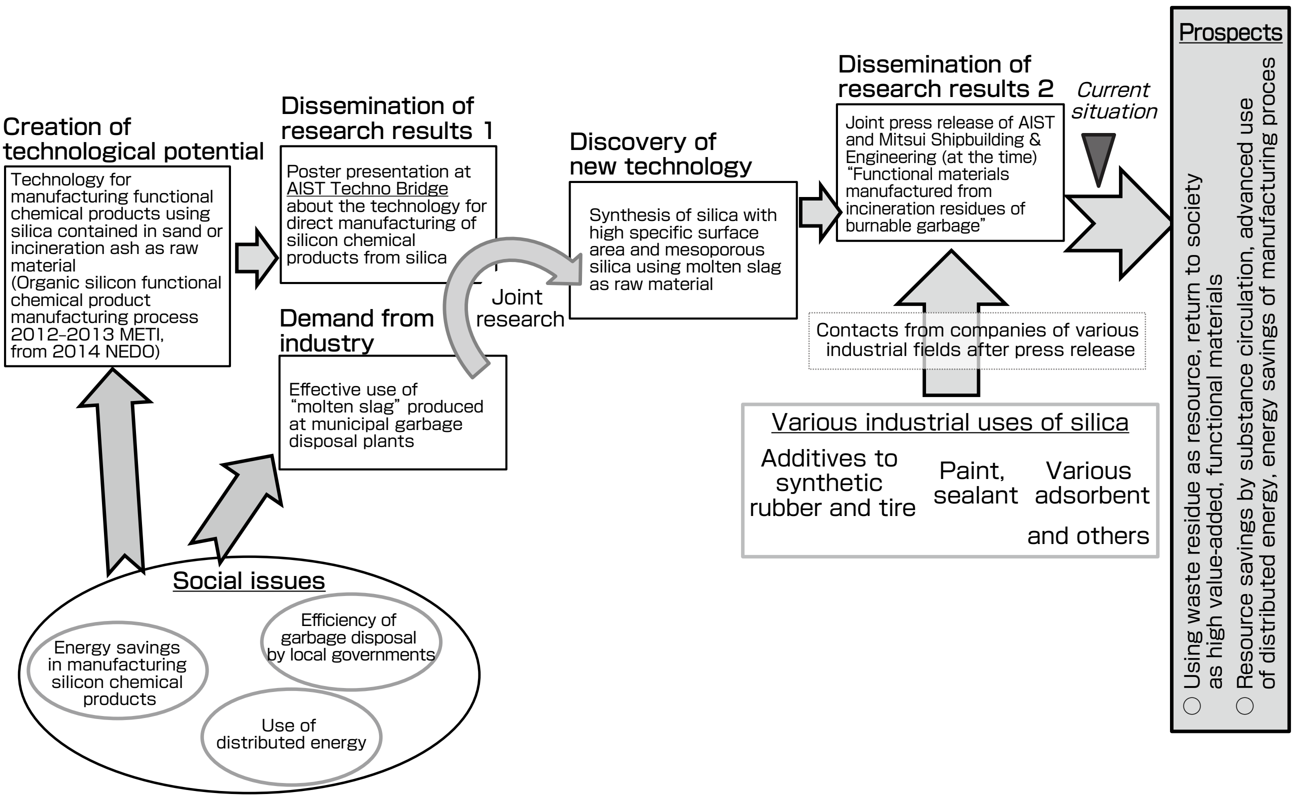

Fig. 1 Scenario of this research and future prospects

2.3 For additional effective use of molten slag

Currently, there are high demands for civil engineering construction materials such as aforementioned road and concrete aggregates due to infrastructure construction for the 2020 Olympics and Paralympics as well as for recovery from earthquake disasters. However, there is concern that the amount of molten slag produced by garbage disposal plants may become excessive against the demand after the Olympics. Therefore, there is demand for technological development to create high added-value materials from molten slag and to enable effective use in wide-ranging fields. Mitsui E&S is an engineering company that conducts business with local governments to construct, operate, and maintain garbage disposal plants. The company has strong interest in developing methodology for effective use of molten slag, and was looking for technological potential at universities and public institutions to solve the problem.

On the other hand, the Interdisciplinary Research Center for Catalytic Chemistry, AIST has been engaging in R&D for various organosilicon materials including silicone that was used in a wide-range of industrial fields in the NEDO Project "Development of the manufacturing process technology for organosilicon functional chemical products" (FY 2012–2021). The authors gave a poster presentation of the research results of the above NEDO Project at the AIST Techno Bridge held on October 22–23, 2015. R&D personnel of Mitsui E&S visited the exhibition and became interested in the content of "Direct synthesis of silicon key chemical products using silica as raw material." They contacted the authors later, and the possibility for the effective use of molten slag and incineration ash that were produced from energy facilities was discussed, and joint research was started. In the following subchapter, we explain the outline of the technological potential created through the results of the NEDO Project that was the preliminary step to joint research.

2.4 Outline of the NEDO Project "Development of the manufacturing process technology for organic silicon functional chemical products"

Silicon is the second most abundant element after oxygen, among the components that comprise the earth surface. In nature, it exists in the form of silica that is the main component of rock and sand. There are many materials containing silicon around us, and it is used in various forms. The organic silicon chemicals represented by silicone and silane coupling agents have excellent physical properties from the viewpoints of heat resistance, weather resistance, chemical resistance, insulation property, and others. They are functional materials used widely in industrial fields such as of automobiles, aerospace, construction, electronics, medicine, cosmetics, and others. A compound called tetraalkoxysilane (e.g. tetraethoxysilane is known by the abbreviation TEOS) is widely used as raw material of inorganic silicon material and is a key substance essential for making protective films for electronic devices, as well as optical materials such as functional ceramics, glass, and synthetic quartz.

Chemical products containing silicon that are used in various materials must be reduced to metallic silicon by making the starting material, natural quartzite, react with carbon at high temperature. This process requires a large amount of electric energy, and at the same time, produces a large amount of carbon dioxide (CO_2). This is the main factor that makes chemical materials containing silicon relatively expensive products, even though silicon is an element that exists abundantly on this planet. There is expectation for the development of technology to directly synthesize silicon-containing chemical products from silica that is reasonably priced and exists abundantly in Japan (Fig. 4).

The authors aimed to develop a new manufacturing method for organic silicon chemical products that did not take the route of metal silicon, and worked on the development of technology to synthesize tetraalkoxysilane directly from silica, and succeeded in achieving a highly efficient reaction process. The characteristic of this technology is that a

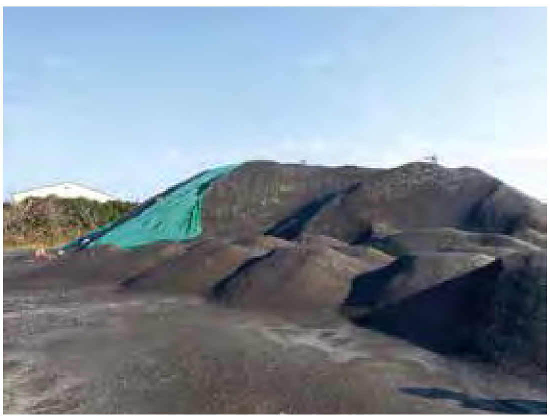

Fig. 2 Storage yard of molten slag

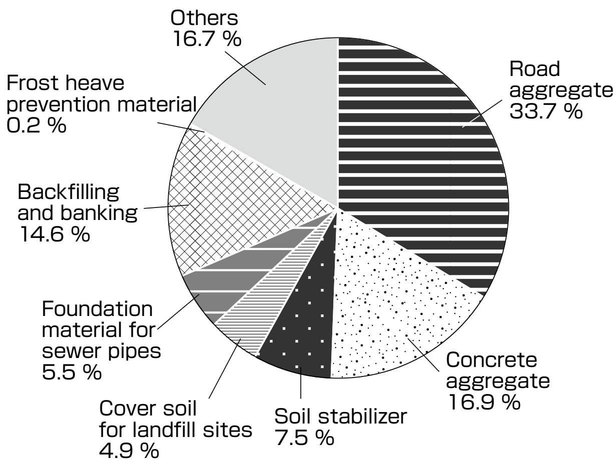

Fig. 3 Current situation of use of molten slag (from garbage and sewage sludge)[2]

Table 1. Results of direct synthesis of tetraethoxysilane from various natural materials

Raw material containing silica	Purity of silica in raw materials	Tetraethoxysilane yield
Sand[a]	90 %	51 %
Incineration ash from chaff	93 %	78 %
Incineration ash from straw	84 %	72 %
Industrial by-products[b]	95 % or more	72 %

a: Obtained by crushing siliceous shale from Natsudomari Penninsula, Aomori Prefecture (provided by Asaka Riken Co., Ltd.)
b: Silica produced as by-product of synthetic quartz manufacturing (provided by CoorsTek K.K.)

process was designed to remove water continuously during the reaction by installing a water removal unit in the reaction system. This enables conversion to useful chemical products through direct reaction, even for SiO_2 that is a substance for which chemical conversion is normally difficult since it is an extremely stable oxide. Since the molecular sieve, or the inorganic desiccant used here, is solid, it can be easily recovered after the reaction and used repeatedly after heating and evacuation. This is advantageous for reducing manufacturing costs (Fig. 5).[3]–[6]

As a starting material containing silica, the following materials were used: sand (obtained by crushing siliceous shale from Natsudomari Penninsula, Aomori Prefecture), incineration ash (remains of burning chaff and straw), and an industrial by-product (silica produced as a by-product

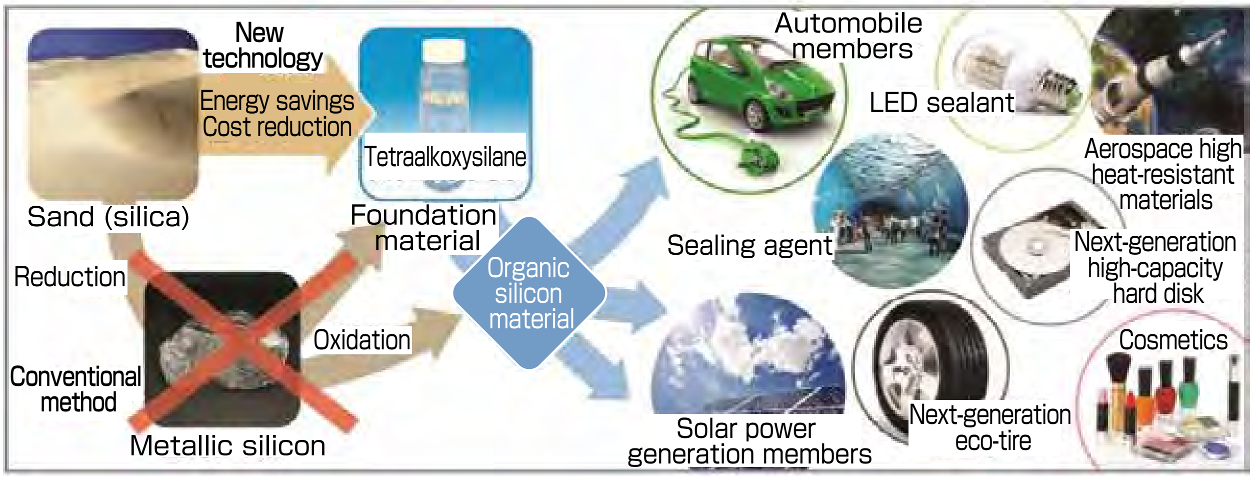

Fig. 4 Manufacture of organic silicon material from sand (SiO_2), and various product groups that contain organic silicon materials

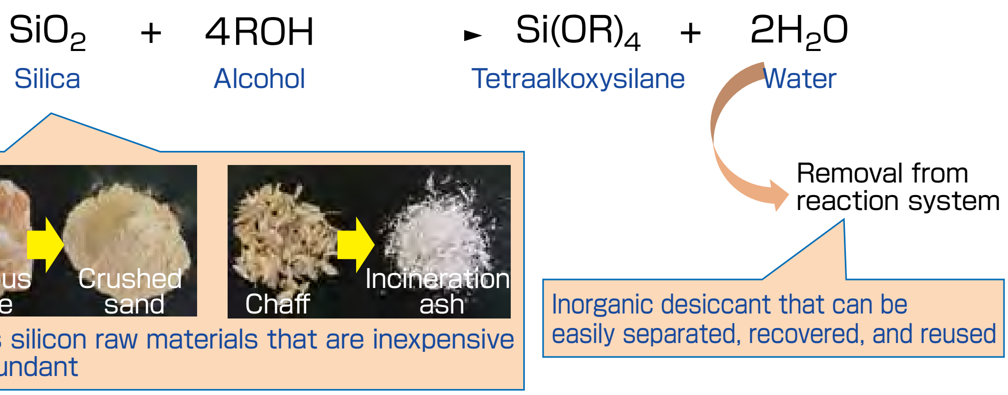

Fig. 5 Direct synthesis of organic silicon material using silica as raw material

in manufacturing synthetic quartz). Ethanol and catalyst potassium hydroxide were added, the mixture was heated along with molecular sieve 3A (pore diameter 0.3 nm), a type of zeolite, as an inorganic desiccant, and the reaction was conducted for 3 hours. Table 1 shows the results of the reaction. TEOS was generated at a yield of 51 % based on the silica content. The ashes remaining after burning chaff and straw that are underused resources of agricultural by-products have relatively high silica purity, and TEOS was obtained at a yield of 72–78 % when these materials were used for synthesis. The by-products of synthetic quartz manufacturing were recovered and used as the starting material, and TEOS was obtained at a 72 % yield.

The point of difficulty in the development process of this technology was the shift in the idea that allowed molecular sieves to be used in this reaction and the design of the reaction apparatus. The most ideal process to synthesize tetraalkoxysilane from silica and alcohol can be described by an extremely simple chemical reaction equation shown at the top of Fig. 5. However, in practice, this reaction is governed by chemical equilibrium, and since silica is a thermodynamically stable substance, the produced tetraalkoxysilane and water react, and a reverse reaction in which tetraalkoxysilane returns to silica and alcohol occurs much more easily. Thus, it is difficult to obtain tetraalkoxysilane at a good yield. To shift the chemical equilibrium to a desired direction, a dehydration method by which produced water is successively removed from the reaction system is the key. Molecular sieves are desiccants used universally, and normally they are used in direct contact with targets (liquid or gas) to be dehydrated. While having powerful desiccant capabilities, they can also be relatively easily reversed and reused, and high efficiency can be expected if they can be used. However, molecular sieves are crystallized alkaline metal aluminosilicate (alkaline metal salt that is composite of alumina and silica), and a silica unit exists within its structure. Therefore, if molecular sieves are directly introduced to the reaction system whose purpose is to dissolve silica, the structure of the sieve will disintegrate. Therefore, at the beginning of the research, we thought we could not use the sieves for this reaction, and we searched for an organic desiccant that could be directly placed in the reaction system.[4]

However, with the progress of the NEDO Project, we became aware of the necessity of developing a simple process that allowed easy separation from the target substance. The desiccant used had to be easily recoverable and reusable, in order to be feasible as the new reaction process was moved up from a laboratory scale to an industrial scale. Therefore, we shifted our way of thinking to how to use a molecular sieve that tended to disintegrate in our reaction condition. We worked on the reaction apparatus, and as shown in Fig. 6, the vessel for silica and alcohol to react under the presence of a potassium hydroxide catalyst (lower part) and the vessel in which a molecular sieve functioned as a desiccant (top part) were separated. In this reaction apparatus, alcohol and water vaporized in the lower vessel, moved to the upper molecular sieve, and only the water was absorbed and removed. Therefore, the potassium hydroxide catalyst that promoted the breaking of the Si-O-Si bond of silica did not come in contact with the molecular sieve, and the water could be removed successively without the disintegration of the structure.

3 Manufacturing of high added-value materials from molten slag raw materials

3.1 Technology to manufacture silica with high specific surface area from molten slag

Mitsui E&S focused on the fact that the research results of the aforementioned NEDO Project involved the "advanced use of silica that was the main component of molten slag" while pointing to the attainment of green sustainable chemistry (GSC). Therefore, the company started joint research with AIST to aim for further effective use of molten slag, particularly for its use in functional materials with new

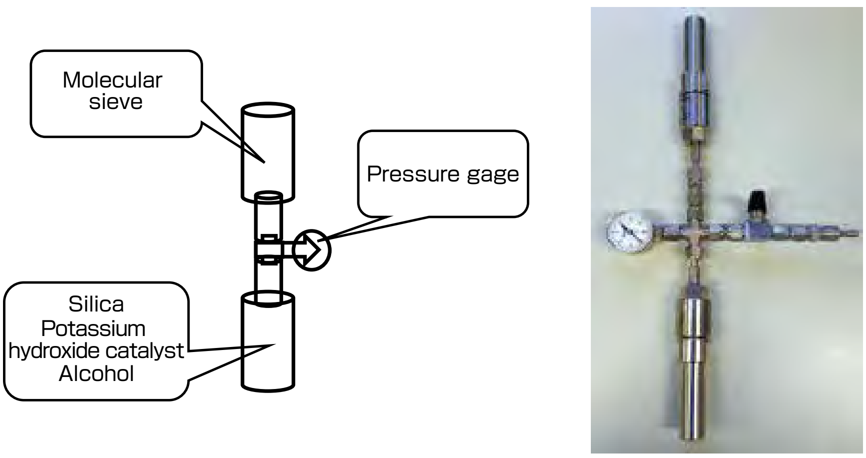

Fig. 6 Schematic diagram of reaction vessel for tetraalkoxylsilane and its photograph[3]

added value for the “arterial industry,” rather than the use in materials such as concrete aggregate for the so-called “venous industry.”

It was newly found that by chemically treating the molten slag produced from municipal garbage disposal plants using acidic solutions under certain conditions, the silica component in the molten slag precipitated as white solids. When this white solid was recovered by filtering or other methods, silica with purity of 93–98 % could be easily obtained (Fig. 7).

The estimated mechanism of this reaction is examined. As explained in Subchapter 2.2, the main components of molten slag are SiO_2, CaO, and Al_2O_3, and the silicon components are thought to exist as “calcium salt of composite oxide of silicon and aluminum” rather than in a pure silica form. This calcium salt is thought to go through a certain process. After undergoing neutralization by acid, aluminum and calcium become soluble in an acid aqueous solution as ions, and silicon increases molecular weight by a dehydration-condensation reaction through a catalyst action of acid, after undergoing a low molecular silica condition called silica sol or orthosilicate. Then it precipitates as insoluble white solids as silica undergoes gelling. That is, in this reaction system, acid has two roles: one is to make components other than silica soluble as ions, and the other is to act as a catalyst for silica production by a sol-gel reaction. Since condensation occurs after the sol state in which silica has low molecular weight and is highly dispersed, the silica that is ultimately obtained by gelling is expected to have a high specific surface area.

The results of actual nitrogen gas adsorption measurement showed that the calculated specific surface area was about 600 m^2/g, and this was equivalent or higher than that of synthetic silica material that was commercially available as high specific surface area material. Therefore, it is expected that silica with high specific surface area obtained from molten slag using this technology will have various usages as adsorbents, additives for tires and synthetic rubber, catalyst carriers, in cosmetics, as abrasives, and others, in which synthetic silica is currently used in industry. Also, silica with high specific surface area will have many reaction points that can come in contact with reaction targets such as alcohol or alkaline catalysts. Therefore, when considered as a raw material for chemical reactions, it is expected to be useful in manufacturing tetraalkoxysilane mentioned in Subchapter 2.4.

Currently, silica with high specific surface area used widely in industry includes fumed silica manufactured by a gas phase reaction of silicon tetrachloride in high-temperature hydrogen fire, or precipitated silica (sometimes called white carbon) manufactured by a liquid phase reaction of sodium silicate and sulfuric acid. These manufacturing methods consume much energy for silicon tetrachloride and sodium silicate, and use raw materials manufactured by processes that require a certain degree of cost. On the other hand, the newly developed technology allows the use of residues that are generated irreversibly while disposing burnable garbage produced in our daily lives, and is overwhelmingly inexpensive at about 200 yen/ton even for those with qualities that satisfy the JIS standard. Therefore, compared to the conventional processes, it is expected to contribute to energy savings during silica manufacturing, reduction of carbon dioxide emission, and cost reduction of silica products.

3.2 Technology to manufacture ordered nano-mesoporous silica from molten slag

Recently, a porous silica material with ordered and uniform pore sizes of 2–50 nm (mesopores) called mesoporous silica is gaining attention. Mesoporous silica is generally synthesized by using a structure made by self-assembly of surfactants in solution as the mesopore template and forming silica around this template by a sol-gel reaction. Mesoporous silica has an ordered structure and has uniform nanospace within the material, and it is expected as a highly selective reaction field that cannot be realized by existing porous materials. It is a high added-value material for which research is conducted around the world for its application as a high

Fig. 7 Raw material molten slag (left) and synthesized silica with high specific surface area (right)

performance humidity control agent, a drug transfer system, or a catalyst carrier.[7]

Silica obtained from molten slag mentioned in the previous subchapter has high specific surface area but does not have a pore structure. Therefore, we engaged in the synthesis of mesoporous silica for the purpose of developing technology to convert molten slag into a higher performance, higher added-value material.

The chemical treatment to obtain silica with high specific surface area shown in the previous subchapter was conducted in a condition in which surfactants that acted as templates to make the pores coexisted. The white solids obtained was fired at 550 °C to burn away the template, and we were able to obtain mesoporous silica with ordered nano-size pores. The specific surface area of mesoporous silica produced from molten slag was 675 m^2/g and the average pore size was 9.2 nm. The electron microscope photograph of this mesoporus silica is shown in Fig. 8. The already known mesoporous silica was synthesized from highly pure and relatively expensive silicon raw materials such as colloidal silica and tetraalkoxysilane. On the other hand, molten slag was not pretreated in this technology and contained large amounts of impurities such as calcium and aluminum. We succeeded in synthesizing the material with pores and specific surface areas similar to the conventional mesoporous silica by maneuvering the reaction time and temperature and finding the condition in which self-assembly of the template surfactants progressed.

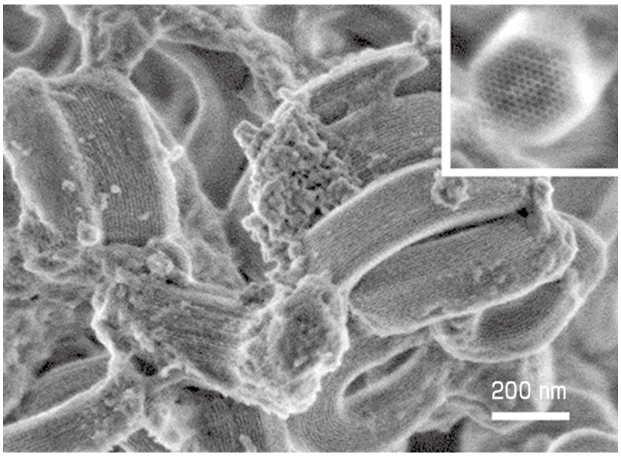

Fig. 8 Electron microscope photograph of mesoporous silica synthesized from molten slag

Both AIST and Mitsui E&S published press releases of the results of this joint research on July 25, 2017 (Fig. 9).[8][9] We were contacted by companies of various industries including chemicals, nonferrous metals, ceramics, traders, and others, and we are now preparing for supplying test sample of silica with high specific surface area to users.

4 Future prospects

The technology developed in this research can expand the advanced use of molten slag, as it leads to wide-ranging use of silica with high specific surface area that can be made from molten slag whose current use is limited. In the future, we aim to improve the manufacturing process, work on upsizing the manufacturing scale, and try for practical utilization in a few years. Through practical realization of this technology, we hope it leads to such outcomes as added value through effective use of waste residue and resource savings through substance circulation.

5 Acknowledgement

The research pertaining to the direct synthesis of organic silicon material using silica as raw material in this paper was conducted as part of the NEDO Project “Development of the manufacturing process technology for organic silicon functional chemical products” (FY 2014 up to the present).

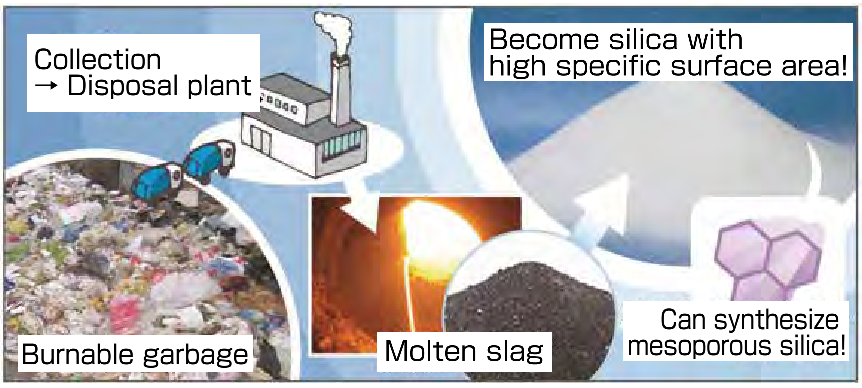

Fig. 9 Conversion of molten slag, incineration residue of burnable garbage, into high added-value material

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Discussions with Reviewers

1 Overall

Comment (Naoto Kobayashi, Waseda University)

This paper presents highly significant research result from the perspectives of practical realization through collaboration with industry, as well as technological development and its application, aiming at the effective use of molten slag produced from garbage incineration plants. Also, the concept of conversion from “venous industry” to “arterial industry” is very attractive. On the other hand, the details of the elemental technologies and their mutual relationships were not clear in the first draft, but this became clear in the final draft, and now the whole story is easier to understand.

Comment (Keiichi Ikegami, AIST)

This paper describes the development of technology to generate high performance materials that are made from incineration residues that have negative value (requires disposal cost). These materials are valuable in arterial industry, rather than in venous industry. Unlike conventional PET recycling and precious metal recovery that involve sorting and obtaining products with high purity, the innovation here is that smart synthesis is carried out based on the chemical equilibrium theory. As the authors write, it is an excellent example that shows that “when research results progress to the next stage, collaboration with human resources with different specialties is important,” and therefore, this paper is appropriate for publication in *Synthesiology*.

2 Collaboration with human resources of different fields

Comment (Keiichi Ikegami)

"When research results progress to the next stage, collaboration with human resources with different specialties is important" is indeed an important point. I think it will increase interest of the readers if you explain more specifically "the collaboration with human resources with different specialties" in each subchapter.

Answer (Norihisa Fukaya)

I added a text in "Subchapter 1.2 Scenario of this research" as follows: "Since the field of specialties were totally different among the specialists who had the knowledge of plant operation for waste disposal plant at Mitsui E&S, the researchers working on chemical reaction and molecular design at the Interdisciplinary Research Center for Catalytic Chemistry, AIST, and the researchers studying chemical engineering or surface chemistry at the Research Institute for Chemical Process Technology, AIST, there was no opportunity for these people to directly exchange opinions in usual activities such as at academic meetings or at paper presentations. … Therefore, events such as the Techno Bridge Fair at which AIST's technological potential is introduced to the people of industry are important, so they can get an overview of the research conducted at AIST and to transmit the results widely and systemically. Looking back, it can be said that the breakthrough point of this research was the matchmaking between the 'seeds' and 'needs' of the specialists from totally different fields, and the fact that they were able to start joint research to tackle new topics."

3 Relationship of the elemental technologies

Comment (Naoto Kobayashi)

The intent of "2.4 Outline of the NEDO Project 'Development of the manufacturing process technology for organic silicon functional chemical products'" is the production of tetraalkoxysilane from silica, and it does not necessarily contribute to the production of "silica with high specific surface area" that was included in the subtitle of the first draft. "3 Manufacturing of high added-value materials from molten slag raw materials" is the only contribution to the subtitle. If you intend to describe the results of both elemental technologies, you should change the subtitle and the research objective, and if the former is contributing greatly to the latter, you should describe their relationship.

Answer (Norihisa Fukaya)

As you indicated, I changed the subtitle so the focus will include the course from molten slag to tetraalkoxysilane (key material of silicon chemical industry). To clarify their relationship, I added a text to the end of Subchapter 3.1 as follows: "Also, silica with high specific surface area will have many reaction points that can come in contact with reaction targets such as alcohol or alkaline catalysts. Therefore, when considered as the raw material for chemical reactions, it is expected to be useful in manufacturing tetraalkoxysilane mentioned in Subchapter 2.4." (Although we do have experimental evidence, only the concept is outlined due to a nondisclosure agreement.)

4 Details of the strategy

Comment (Keiichi Ikegami)

You write, "It was newly found that by chemically treating molten slag produced from municipal garbage disposal plants using acidic solutions under certain conditions, silica components in the molten slag precipitated as white solids." This seems to come up rather abruptly. Perhaps you are not able to disclose the entire process, but can you explain a bit more about how you made this discovery and what strategy you took to achieve this?

Answer (Norihisa Fukaya)

I added the discussion on the reaction mechanism for this chemical treatment at the beginning of Subchapter 3.1: "The estimated mechanism of this reaction is examined. … That is, in this reaction system, acid has two roles: one is to make components other than silica soluble as ions, and the other is to act as a catalyst for silica production by a sol-gel reaction. Since condensation occurs after the sol state, in which silica has low molecular weight and is highly dispersed, the silica that is ultimately obtained by gelling is expected to have a high specific surface area."

5 Advancement of the technology

Comment (Keiichi Ikegami)

You advance the technology further in Subchapter 3.2, but what were the demands and issues in this case? In other words, why did you aim for porosity, not just the high specific surface area? (The improvement of specific surface area is little over 10 %, but can you expect dramatic increase in sales prices if you achieve a porous material?) Also, you write that the chemical treatment is conducted in acidic solution, but did you have any technological barriers in getting the surfactant to work in increased ionic strength?

Answer (Norihisa Fukaya)

I added the expectation about mesoporous silica materials and the citations for such expectations, to clarify the topics and demands for mesoporous silica. I also added the comment about the necessity of know-how to get the surfactant to function when using molten slag as the raw material.

Development and commercialization of laser inspection system to detect surface flaws of machined holes

Saburo OKADA[1*], Osamu NAKAMURA[1] and Yasufumi ESAKI[2]

[Translation from *Synthesiology*, Vol.11, No.3, p.137–147 (2018)]

An indispensable aspect of manufacturing is the external inspection of all product parts. For example, in the manufacturing of cars, autonomous inspection technology is required to detect minute flaws on glossy or mirror surfaces, which are easily overlooked by visual inspection. In this paper, we report on the history, significance, and future development of an innovative defect inspection system, "ANALYZER," which has been developed and commercialized. This system utilizes AIST technology—optical diffraction by semiconductor laser—to realize accurate, autonomous inspection of inner wall surfaces of high quality machined holes of various sizes.

Keywords **:** Flaw defects inspection system, semiconductor laser, diffracted light, optical fiber, cylinder bore

1 Introduction

To guarantee that the parts assembled in the automobiles will perform and function as designed, external inspection of the parts is essential. While there is much money spent on automating inspection in anticipation of future labor shortage, there are many areas that remain dependent on visual inspections, and there is strong demand for high-performance automatic inspection technology.[1] Visual inspections are carried out for the inner wall surfaces of machined holes with various diameters of cylinder bores, automatic transmission valve bodies, hydraulic cylinders, and others that are important safety parts of automobiles, since they have particularly strict inspection standards. As no oversight is tolerated, there is demand for high-precision and high-speed automated inspection technology that can detect minute flaws and defects of about 0.1 mm. Systems with various methods have been developed such as eddy current, camera imaging, and laser reflection, but none attained the level that could satisfy on-site demands. Okada *et al.* have engaged in R&D of advanced industrial measurement systems using semiconductor lasers that are small, lightweight, and easy to handle, and developed new devices that could measure glossy or mirror surfaces that were difficult to measure with conventional measurement technology. Utilizing this experience, and collaborating closely with regional companies, AIST and Sigma engaged in the development of a system to conduct high-speed and high-precision inspection for minute flaws and defects on the inner wall surfaces of machined holes with various diameters, and finally succeeded in developing and commercializing a laser defect inspection system.

In this paper, we explain the development of a system that enables measurement of glossy and mirror surfaces using semiconductor lasers that is the core technology in the development of a laser defect inspection system. Then, we describe the course and the collaborative activities for developing and commercializing the original laser defect inspection system for specific targets including the exterior/interior surfaces of cylinders and the interior of machined holes with various diameters, starting with the surface defect inspection of high-grade steel sheets that was initiated after technological consultation from a regional company. Then, the significance of this development and prospects for the future will be addressed.

2 Course toward development of laser defect inspection system

Ever since 1980, semiconductor laser elements that irradiated at a wide range of wavelengths from ultraviolet to near-infrared light were developed successively, and industrial application of laser beams expanded rapidly. In the field of industrial measurement, in place of the conventional large gas lasers that were vulnerable to impact and vibration, there was growing demand for measurement technology using semiconductor lasers that were small, lightweight, and easy to handle. Based on this background, Okada *et al.* were motivated to engage in R&D for new industrial measurement technology that efficiently utilized the characteristic of semiconductor lasers.

First, to conduct stable and highly precise measurement of molds and parts that are glossy, high-grade, and with free-form surfaces, Okada engaged in the development of form

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measurement based on a coaxial linear displacement method, collaborated with Osaka University. The coaxial linear displacement method has advantages of being less likely affected by specular reflection light, relationship between displacement and output being linear, and precision being unchanging in all measurement ranges. Therefore, there was much expectation for realization, but, as shown in Fig. 1, the realization was hampered by the reduction of precision due to speckles[Term 1] that were characteristics of laser beams.[2] To solve this problem, Okada *et al.* used a high-density line sensor instead of an area sensor in the photoreceptor as shown in Fig. 2, and created a unique mechanism for rotating the sensor. It was demonstrated that the speckles could be reduced greatly by rotating the line sensor at 200 rpm and conducting space averaging. That is, as shown in Fig. 3, the speckles reduced due to the rotation of the line sensor and the image quality improved, and this allowed the measurement of shapes at precision within 0.1 mm in the measurement range of 150 mm. The light at the end of the tunnel for the road toward realization could be seen.[3]

Next, Okada worked on the development of a noncontact 3D measurement device for mirror surface objects that were more difficult than glossy surfaces. Since mirror objects totally reflected laser beams and specular points could not be seen at all, it was extremely difficult to measure surface forms, and while measurement could be done for flat surfaces, there was no measurement device that could measure curved surfaces. Therefore, Okada devised a method for calculating the 3D coordinates of specular points based on a ray tracing method, by capturing the laser reflection light in multiple positions in 3D space by rotating several position sensitive detectors (PSD) arranged in a dome shape. Figure 4 shows the appearance of the mirror-surface object measurement device[5] that was developed and prototyped. By capturing reflected laser beams in two places of the 3D space by arranging two sets of four PSDs unevenly in a vertical direction, an equation for laser beams that pass through two points in the 3D space was determined. Then, form measurement became possible by setting the intersection point with the irradiation light as a virtual reflection point.[4] A patent was filed for this technology and was registered as intellectual property, and was selected as a notable invention by the Agency of Science and Technology in 2000 (Patent No. 317857, 1999.2).

As Okada was working on the R&D for new measurement technology to utilize semiconductor lasers in industrial measurement, Okada was consulted by a local steel sheet manufacturer about a device to inspect minute flaws and defects in rolled steel sheet surfaces with high glossiness. This launched us into the development of inspection technology using lasers. The requests from the steel sheet manufacturer were the detection of micro-defects of micron order on the surfaces of high-grade rolled steel sheets, the separation of defects and roll marks, and the distinction of detected defect types.

The newly developed laser defect inspection system[5] is shown in Fig. 5. The point of development is the structure for measuring light intensity distribution of the reflected scattered light and diffracted light using a planar photodetector placed at a focal position by gathering all the reflected light within the measurement range to a focal point, and using parabolic cylindrical mirrors in the photoreceptor system in addition to

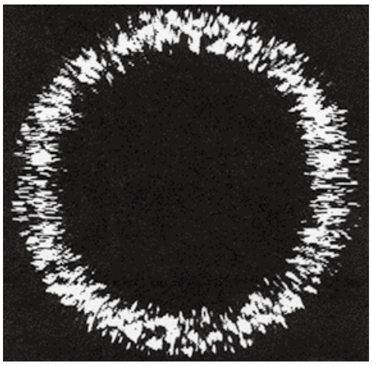


Fig. 1 Laser beam ring image by area sensor

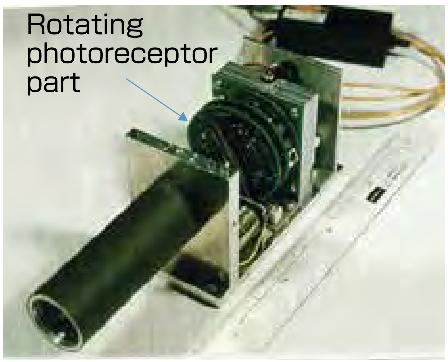


Fig. 2 Developed and prototyped measurement system

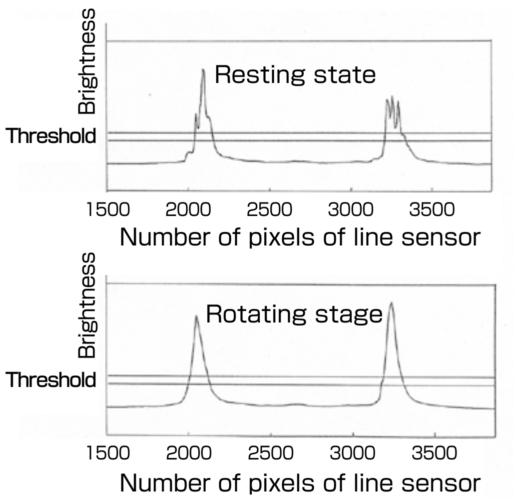


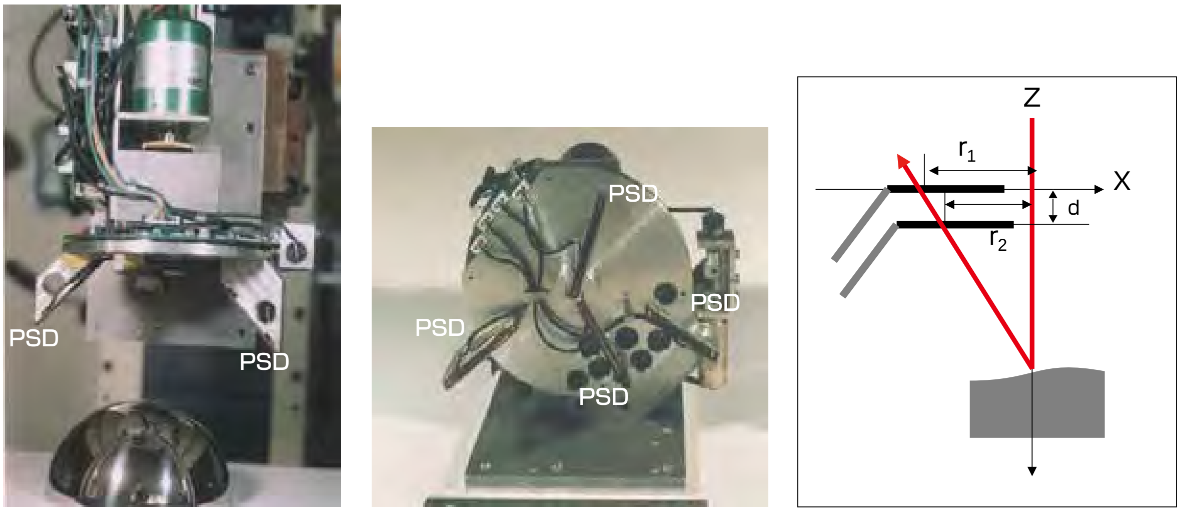
Fig. 3 Effect of line sensor rotation

the phototransmitter system of laser beams. As shown in Fig. 6, the multi-segmentation planar photodetector is made by bundling about 3,000 optical fibers of a diameter of 0.5 mm in a semicircular form, and these are divided into four in a radial direction, and into 12 in a circumferential direction. Then light intensity entering a total of 48 blocks is photoelectrically converted using photodiodes. This detector has rougher resolution compared to camera images but is adequate in grasping the characteristics of the reflected light patterns, and also has the advantage that the measurement time per point is high speed at 1 msec or less, and the data volume can be reduced to 1/500 or less.

Of the types of defects produced in steel sheet manufacturing shown in Table 1, the most frequently found scratches and abrasions are shown as examples in Fig. 7 that shows light intensity distribution by LED that was measured by a multi-segmentation planar photodetector and camera images of their reflected scattered light. It was demonstrated that the types of defects could be identified by the multi-segmentation planar photodetector. Here, linear light in the vertical direction is the diffracted light produced by roll marks, and this is detected on the entire surface of rolled steel sheets. However, since the roll direction is only in one direction in the steel sheet rolling process, the diffracted light of roll marks is produced only in a vertical direction, and this is discriminated from defects by removing the diffraction light that occurs in this direction from the inspection.

Table 1. Type of flaws

Form of flaw	Name of flaw
Pointlike defect	Roll mark
Linear defect	Scab, sliver, scratch
Distributed defect	Rust, contamination, abrasion



Position of PSD

Measurement principle

Fig. 4 Measurement system for curved mirror objects

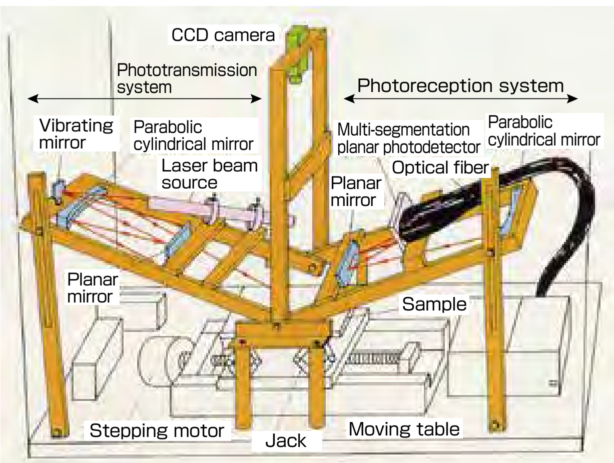


Fig. 5 Laser defect inspection system for flat sheets

Fig. 6 Multi-segmentation planar photodetector

This technology has been filed for patent and registered as intellectual property (Patent No. 2073658, 1996.7).

3 Development of defect inspection system for inner wall surfaces of machined hole

What triggered machined hole inspection was technological consultation from an automobile parts manufacturer in Hiroshima around 1998, about the inspection of minute flaws and defects in the inner walls of automobile hydraulic master cylinders that were parts manufactured by the company. The target was a cylindrical part with an inner diameter of 25 mm, depth of 150 mm, and a mirror-polished inner wall. Since it was hard to view the inner wall by visual inspection and minute defects were often missed, Okada decided to conduct the inspection using laser beams. Since the target part was cylindrical and rotatable, the device shown in Fig. 8 was made as a prototype.[6] If there were no defects when the finely focused laser beam is irradiated onto the inspected surface, the laser beam reflects off without scattering. On the other hand, if there was a defect, the light that hit the defect scatters widely outside the specular reflection light. We realized that if optical fibers were installed in a position in which specular reflection light could be received and a position in which only scattered light could be received, the two lights could be separated. Therefore, the specular reflection light and scattered/diffracted light were separately received with optical fiber bundles with a diameter of 0.5 mm arranged along the circumference of two concentric circles of a diameter of 5 mm and 15 mm from the center of the reflected laser beam.

Figure 9 shows the panorama images of light intensity data that are shown in a circumferential direction and sampled at 0.2 mm intervals. The image in (a) is the light intensity of the specular reflection light and (b) is the light intensity image including the scattered and diffracted light. The (a) image clearly shows spiral indentation defects caused by broken cutting bites, and (b) shows a scratch. It was shown that various defects could be detected at high sensitivity by using specular reflection light and scattered light. The black and grey holes are oil holes and are not defects.

Around the same time, President Shitanaka of Sigma Corporation, an automobile parts manufacturer in Kure City, saw the laser defect inspection system that was exhibited at a patent fair. He became interested in semiconductor lasers and requested whether this device could be used for external defect inspection for mass-produced automobile parts with cylindrical shapes. Sigma mass produced small automobile parts and was working on automating inspection with the aim of zero shipment of defective products. It was looking

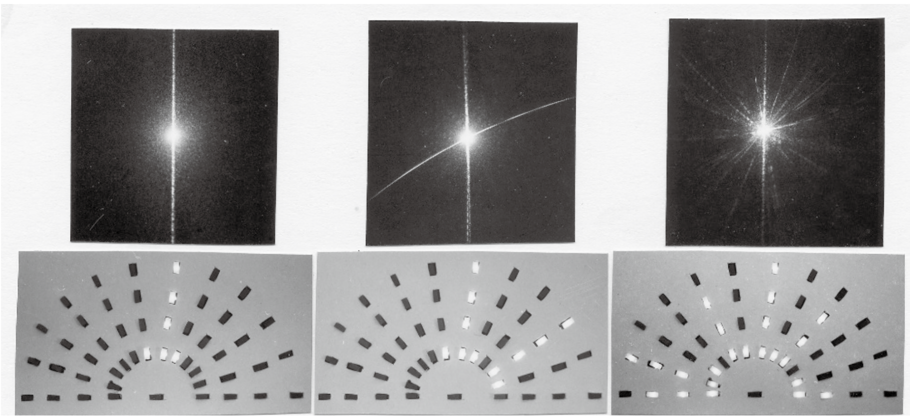


Fig. 7 Differences in diffraction pattern by flaw types and examples of measurement by multi-segmentation planar detector

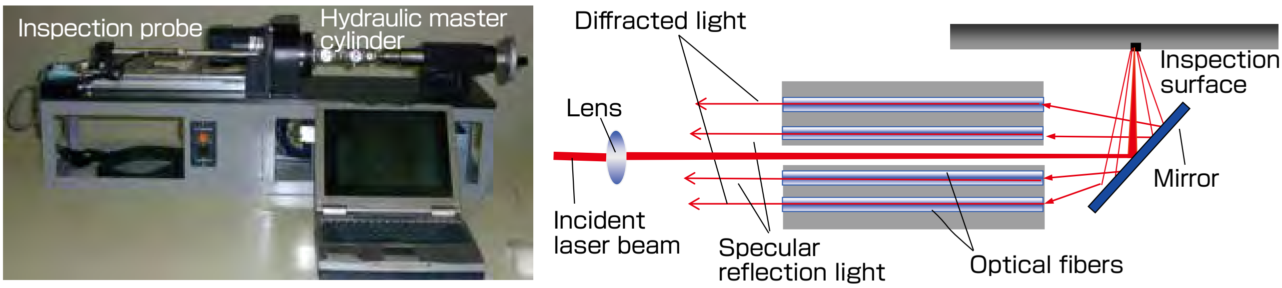


Fig. 8 Inspection system for inner wall surfaces of machined holes

into introducing a laser defect inspection system that could inspect small defects.

Joint research was started in 2000, and the laser defect inspection system for external defects of cylindrical parts was jointly developed by AIST and Sigma in 2002. Figure 10 shows the device. Since the external inspection system using laser beams exceeded expectations in small defect inspection, President Shitanaka proposed to start a business to commercialize and sell the laser defect inspection system that was not yet known in society, and to jointly develop a new laser defect inspection system for the inner surfaces of cylinder bores for automobile manufacturers.

4 Acceleration of R&D through the establishment of collaborative research unit

In 2001, research institutes under the Agency of Industrial Science and Technology, Ministry of International Trade and Industry underwent a major re-organization into the

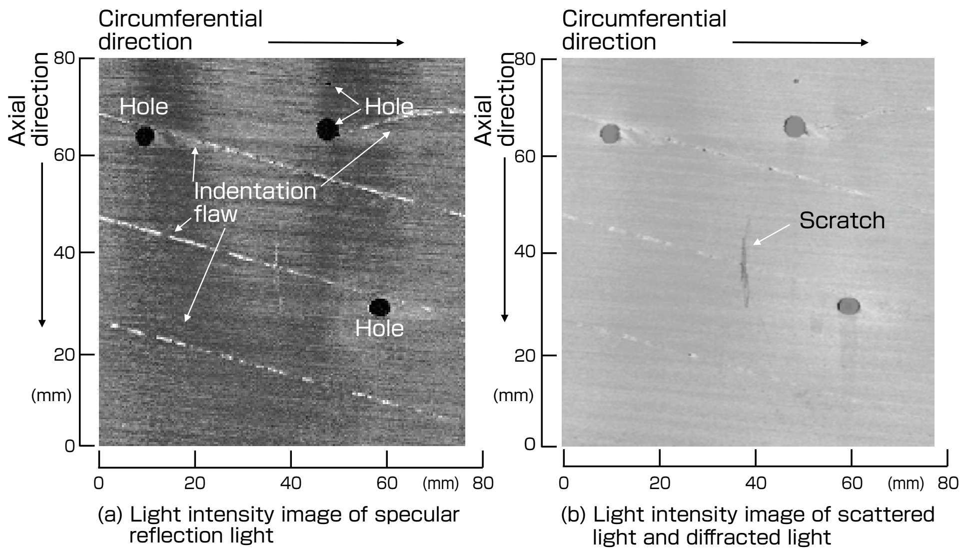


Fig. 9 Examples of inspection images of inner wall surfaces of machined holes

Fig. 10 Inspection system for outer surface flaws of cylindrical automobile parts

National Institute of Advanced Industrial Science and Technology (AIST). In 2003, AIST Chugoku set a policy of placing importance on biomass research, the researchers for manufacturing were transferred to other centers, and we stood at a major crossroad of whether to continue or discontinue the R&D for laser defect inspection system right before its realization. When Okada *et al.* were seeking a solution, President Shitanaka of Sigma who was certain about the future of laser defect inspection strongly requested the continuation of the development, and he was also willing to provide research funds. After discussing with Director-General Yabe (at the time) of AIST Chugoku and the researchers who were transferred to Tsukuba, AIST agreed to continue the R&D based on the funds provided by the company after setting up a collaborative research unit that would be the base of R&D at the Industry Academia Government Collaboration Promotion Division, AIST Chugoku, and we applied for its establishment. There were many conditions for application: there must be requests from many companies; pure private company funds will be provided for three years to fulfill AIST's rules; there must be a clear research goal, and impact on industry, and the project must be achievable by joint research; and there must be research capacity at AIST. The hurdles to clear were high, but the establishment of a collaborative research unit for laser application functional diagnosis was accepted for three years starting in 2004.

The research goal was set as the R&D and product realization of a laser defect inspection system for the inner wall surfaces of automobile cylinder bores, to meet the demands from the automobile manufacturers. The eddy current and optical inspection systems for cylinder bore interior that were commercially available at the time did not satisfy the on-site demands, and a higher-performance and higher-functional inspection system was desired. Figure 11 shows the inspection system of cylinder bore interior that was developed and prototyped through one-year joint research with the basic concept provided by AIST. The key of the development is the structure in which the inspection probe rotating at 1500 rpm is lowered at a steady rate along the central axis of a hole, a semiconductor laser beam formed into a true circle of a diameter of 0.1 mm is irradiated perpendicularly onto the wall surface, the specular reflection light, reflected scattered light, and diffracted light from the wall surface are collected by optical fibers arranged in double concentric circles, light intensity is measured by an optical sensor installed at the other end of the optical fiber, and the defects are detected by light intensity change.[7][8] Since the structure of the probe tip greatly affected the performance, much time and effort were needed for repeated experiments done by changing the tip form and end face position of the optical fiber. However, through the efforts of the development personnel at Sigma, the collaborative research unit was able to find the optimal position and form. Also, the probe tip was made removable, and by employing a structure in which the tip could be slid back and forth, the device could measure a wide-range of inner diameters from 40 mm to 150 mm.

Figure 12 shows the light intensity of specular reflection light measured by inner optical fibers, and Fig. 13 shows the image of the light intensity of scattered light measured by outer optical fibers, and the light intensity data of one rotation. The inspection target was a cylinder bore with an inner diameter of 60 mm, and the measurement was done in 0.2 mm intervals in both the circumferential direction and axial direction. The measurement points were about 4,000 points per rotation, 600 lines in the axial direction, and total data volume was 2,400,000 points (5 megabytes). A clear difference in light intensity distribution between the specular reflection light and scattered light images can be seen.

In the two figures, the figures shown at the bottom is the actual value (blue or red line) of each light intensity per rotation, the maximum and minimum of the thresholds automatically calculated using the actual values are shown in yellow and green lines, and the parts that surpass this range at the top and bottom are candidates of defects. The reason the maximum and minimum are set is because the laser beam is scattered and the light intensity is decreased below

Appearance of inspection device | Inspection probe | Probe tip | Probe structure

Fig. 11 Inspection system for inner wall surfaces of machined holes

the minimum for the specular reflection light image in most defects, while he light intensity increases and may surpass the maximum in the case of glossy defects. Similarly, even in the scattered light image, the defect candidates are the parts in which the scattered light surpasses the upper and lower limits. Here, investigation was done for the difference in response against surface contamination and color between specular reflection light and scattered light using a white tape. In the specular reflection light image, the reflection light intensity increased due to the white tape and was judged as a candidate of a glossy defect, but in the scattered light image, the laser beam did not scatter on the tape surface, the light intensity change was small, and it was judged to be not a defect. Also, in the case there are small holes on the surface, both specular reflection light and scattered light decrease, and they can be judged as defects. As it can be seen, the reliability of inspection could be increased using the two inspection images, and it was extremely effective in preventing erroneous judgment.

In order to conduct the collaborative research activities smoothly and efficiently, the collaborative research unit made efforts to maintain the line of communication by holding a progress meeting which the President attended every other month, gave reports on the development status, and opinions were exchanged about the technological issues, the measures, and long- and short-term development processes. Occasionally, the President determined the policy. Also, if any problems arose, emergency meetings were held by the personnel-in-charge, to conduct quick review and changes of

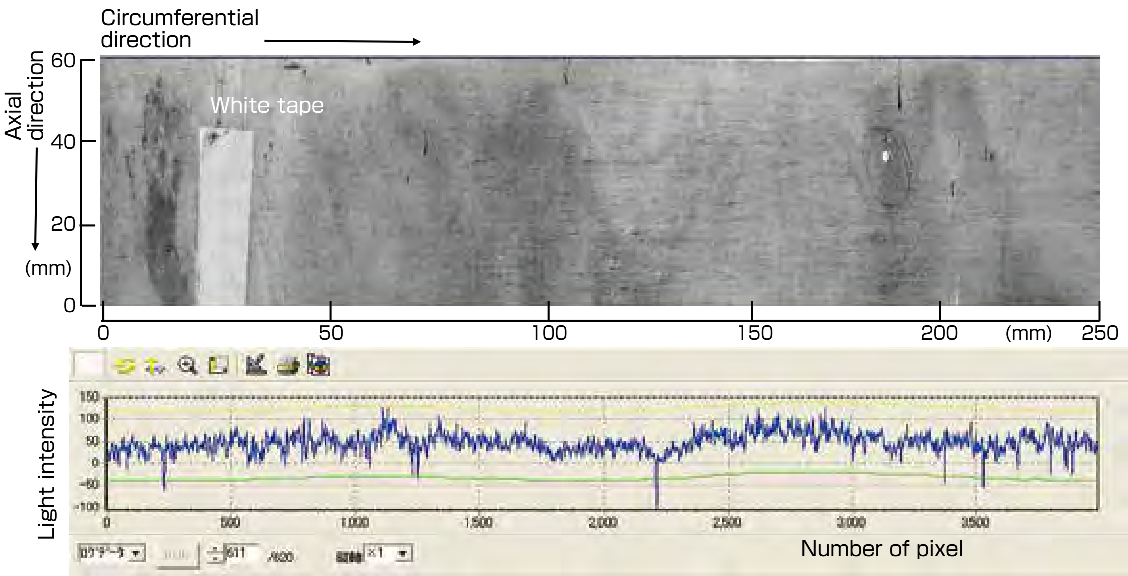


Fig. 12 Light intensity detection image of specular reflection light

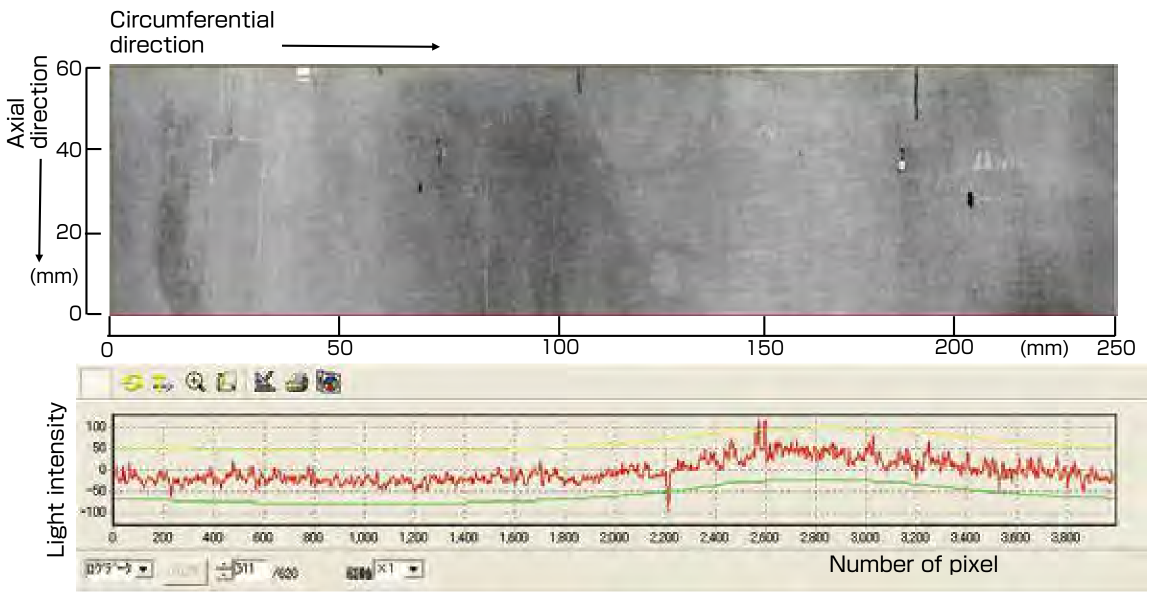


Fig. 13 Light intensity detection image of scattered light

plan. In the case of this project, since the research funds were the matching funds provided by the private company and AIST, necessary items could be procured quickly without concern of the budget, and so the development progressed quickly.

5 Start of laser defect inspection system business and commercialization of ANALYZER

In 2005, Sigma newly established the Division of Laser Defect Inspection System, started sales of the laser defect inspection system, and steadily increased sales from six systems in 2006 to 11 in 2007. To strengthen the sales, Esaki assumed the position of Division Manager. However, hit suddenly by the economic recession due to the Lehman Shock of October 2008, the device did not sell well, the performance of the main business dipped greatly, and the collaborative research unit faced a major crossroad of whether to continue or to retreat from the defect inspection business.

As a measure to discern the future of the project, Esaki set off to visit the companies that purchased the system as well as other machining companies to survey the potential demands for the laser defect inspection system. He found that there was large, unexpected potential demand for the inspection of small holes with inner diameters of 20 mm or less that was outside the target of this system, and that there was need to shorten the inspection time per part from the conventional 30 sec to within 10 sec in order to match the on-site cycle time, because there were many holes that had to be inspected in the small bore hole inspection for automobile parts. These points were noted, and the collaborative research unit proposed the continuation of business to the President and suggested that essential modification be made during the recession, such as reducing the size of the inspection probe and achieving high-speed inspection. These were put to immediate practice after receiving a green light from the President.

For the achievement of high-speed inspection, instead of the belt-driven method using an external motor that was the damper to high speed, we newly developed a hollow motor that enabled high-speed rotation of the probe. As a result, the rotation of the inspection probe was increased to about 10 times or 15,000 rpm, and we succeeded in shortening the inspection time to the target 10 sec or less.

Next, for decreasing the probe size, Sigma newly developed a small diameter probe based on AIST's advice. As shown in Fig. 14, the position of the optical fiber was changed, and the tip was changed from L-shape to straight. The probe diameter was reduced to 6 mm, but to supplement the decrease of receiving light intensity accompanying the reduced number of optical fibers, the photodiode that photoelectrically converted the received reflection light was changed to a high-sensitivity, and high-speed element. By reducing the probe diameter, the obtainable inspection images were only scattered light images, but the level of completion of the product remarkably improved by strengthening the feature value analysis function for the planar form of the abnormal part that surpassed the threshold and by improving the algorithm, and so the defect judgment condition could be adjusted finely.[9]

By 2010, the economy was clearly recovering, and Esaki restarted sales by naming the product ANALYZER (this is a play on words in which a Japanese word, "ana," for holes is used). The orders came in widely from not only the automobile related companies but also companies of various businesses, and the number of sales increased rapidly. Matching users' demands, a standard type of a diameter of 6 mm, a thin type of a diameter of 2.3 mm, and an ANALYZER robot were developed and sold successively. The sales results reached over 200 systems in total.

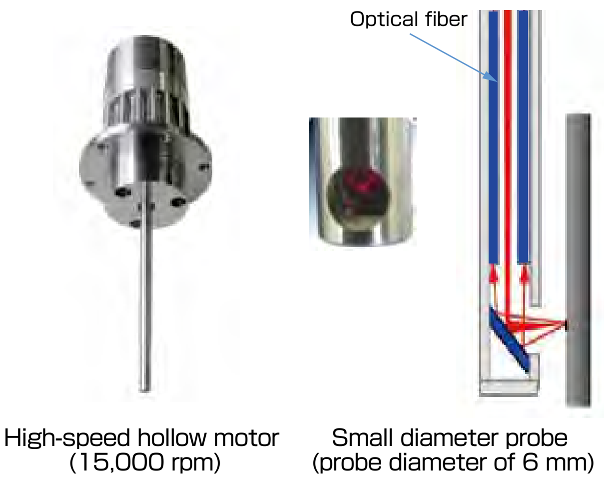


Fig. 14 Modification of inspection probe

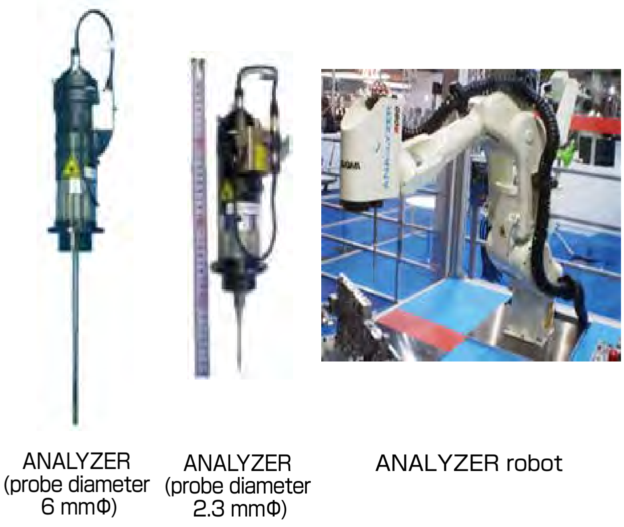


Fig. 15 Example of developed defect inspection system

Table 2. Commercially available inspection systems for inner wall surfaces of machined holes (from HPs of respective companies)

Name of manufacturer	Company A	Company B	Company C	Company D	Sigma Corp
Measurement principle	Eddy current type	Laser reflection type	Camera imaging type	Camera imaging type	Laser reflection type
Characteristic	Two ND probes are arranged facing each other at 180° to detect cavities and cracks, and four air jets are arranged facing each other at 90° to measure interior diameters. It is necessary to change the probe diameter according to the work piece diameter. Applicable diameter of hole: 40 mm or more.	Laser beam is irradiated on one of the two optical fibers, the beam is focused to 50 μm using the lens at the tip and irradiated perpendicular to the interior metal surface, specular reflection light is collected with another fiber, and light intensity is measured by PD. The entire inner surface of a hole is inspected by high-speed rotation of a 45° mirror on the probe tip. The focal depth is shallow since the lens is short-focus. Applicable diameter of hole: 2 mm or more.	The characteristic is to shoot fine images inside cylinders with a high resolution area camera and light rotation mechanism in which several mirrors are rotated in synch. The structure is complex. Applicable diameter of hole: 60 mm or more.	The tip of an acrylic rod is machined into an inverted cone shape, the inner wall surface is illuminated with a ring lamp from the other end, an image of the inner wall projected on the conical surface of the tip is shot with a camera, and defects are inspected by image processing. It is compact and lightweight since there is no rotating part. Applicable diameter of hole: 6 mm ~60 mm.	Laser beams are irradiated perpendicularly to the inspection surface using a long-focus lens, part of specular reflection light is irradiated on several optical fibers that are arranged along the circumference at the probe tip, light intensity is measured by PD, and defects are determined by changes in light intensity. Since a long-focus lens has deep focal depth, there is no reduced sensitivity due to displacement. Applicable diameter of hole: 4 mm or more.

Sigma became the top selling company of this niche market of defect inspection systems for interior of holes in Japan, but as shown in Table 2, various hole interior inspection systems with diverse methods were sold in Japan. However, there was no uniformity in performance indication, and the user companies were confused. Esaki felt the need for standardization of defect inspection systems and started activity for standardization in Japan. In 2015, a committee for drafting the proposal for standardization was established with the support of the Hiroshima Industrial Promotion Organization. Device manufacturers and user companies were asked to participate, and an application was submitted to the standardization system for the creation of new markets of the Japanese Industrial Standards Committee. The proposal was accepted, and the discussions for standardization are currently in progress.

6 Scenario for road to industrial application of semiconductor lasers

Starting with technological consultation from a local company, the collaborative research unit launched R&D for a laser defect inspection that was demanded by industry. Figure 16 shows the flow of product realization through technological development at AIST and collaboration with Sigma explained above. First, to realize defect inspection for inner walls of small and glossy cylindrical parts, we developed a high-sensitive inspection system that separated and measured the diffracted light unique to laser beams that were produced by defects. However, just before commercialization, continuation of R&D seemed difficult due to the re-structuring of AIST, but the development and product realization of a laser defect inspection system of a probe rotating type were continued by establishing a research organization called a collaborative research unit. However, due to the Lehman Shock that occurred suddenly, Sigma faced a crisis of retreat from business, but the policy was changed to meet the demands for inspection of the interior of small holes. As a result of developing a high-speed laser defect inspection system for holes with small diameters that matched the demands of companies, business expanded rapidly. Currently, utilizing the standardization system for the creation of new markets, domestic standardization is being conducted by calling on others in the same industry, and we are also preparing for international standardization in anticipation of overseas expansion.

There were two large turning points in the development and commercialization of the laser defect inspection system. Difficulties were overcome by the ingenuity of AIST and a local company, and Sigma was able to send out the ANALYZER to the world. The device system was purchased by automobile manufacturers such as Toyota Motor Corporation, Honda Motor Company, Ltd., Mazda Motor Corporation, and others, as well as automobile parts manufacturers including Denso Corporation, Aisin Seiki Co., Ltd., Mitsubishi Heavy Industries, Ltd., and others. Sales are increasing to foreign companies such as Daimler AG.

7 Development of next-generation inspection system through collaboration crossing regional centers

AIST sets its important goal as the development of "technology transfer research" in the 4th medium-term goal period, to clarify the mission for functioning as an institution of technology transfer to create innovation. The regional centers of AIST set the main research themes considering the characteristics of regional industrial clusters, and conduct R&D at the highest level. They also collaborate with the regional economy and industrial bureaus and public research institutions, understand the demand of the small and medium-sized companies as well as regional core companies, and aim to contribute to regional revitalization by conducting technological transfer through involvement of all of AIST.[10]

At AIST Chugoku, " AIST Chugoku Network Club (San'yu Kai in Japanese)" consisting of small and medium-sized companies, major companies, and public research institutions of five prefectures in the Chugoku region and other stakeholders was set up in 2011 for collaborative activities such as network formation, and a scheme[11] to extract issues of companies in the Chugoku region by actually visiting sites

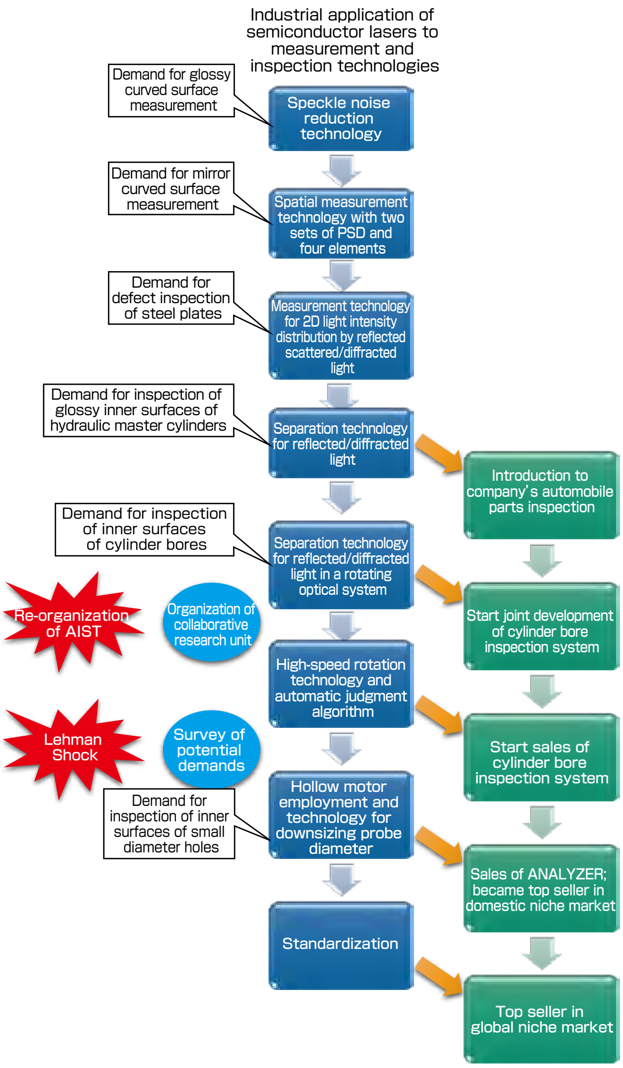


Fig. 16 History of commercialization through technological development by AIST (blue) and collaboration with Sigma (green)

was established.

AIST became estranged from Sigma after the collaborative research unit broke up as the researchers were dispatched to other places. However, in 2011, there was a top meeting between Director-General Nakamura (at the time) of AIST Chugoku and President Shitanaka in which the President requested whether AIST could solve the problem of erroneous or excessive judgment. Nakamura selected a suitable researcher at AIST Kyushu among the researchers of AIST around Japan and the development of a defect inspection system that could tell defects and contamination was started in 2012, and an improved laser defect inspection system was developed and realized with new ideas. With further collaboration with AIST Kyushu, we are working enthusiastically on the development of a next-generation inspection system that is also capable of quality control, and it is expected that we shall have a world-dominating product in not-so-distant future through collaboration that crosses regionality.

Acknowledgement

We shall take this opportunity to thank all the people who were involved in the R&D and commercialization of this laser inspection system. We do hope this paper will contribute to further promotion of future technological transfer activities.

Terminologies

Term 1. Speckle: Dot pattern that appears when coherent light like a laser beam is scattered by an object. It is often the cause of reduced precision in image measurement and is extremely difficult to remove. It is also called speckle noise.

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Completed the master’s course at the Graduate School of Engineering, Hiroshima University in 1974. Joined Government Industrial Research Institute, Chugoku, Agency of Industrial Science and Technology, Ministry of International Trade and Industry in 1974. Engaged in R&D for various hydraulic measurement devices pertaining to the Seto Inland Sea Hydraulic Model. Since 1995, engaged in R&D for measurement devices using semiconductor laser. Coordinator, Industry Academia Government Collaboration Promotion Division, AIST (in charge of metrology standard) in 2005; retired in 2008; coordinator at a public incorporated foundation; and currently working as industry-academia-government collaboration staff at AIST Chugoku from 2014. Doctor of Information Engineering (Kyushu Institute of Technology). In this paper, he was in charge of writing the history of R&D at AIST and the development and realization of the laser defect inspection system.

Osamu NAKAMURA

Completed the master’s course at the Graduate School of Agriculture, Kyushu University in 1979. Assistant professor, Department of Oral Biochemistry, Dental School, Kagoshima University in 1979; Visiting Researcher, Case Western Reserve University in 1989–1991; Research Fellow, Kyushu National Industrial Research Institute, Agency of Industrial Science and Technology, Ministry of International Trade and Industry in 1997; Director, Bioresource Division and Director General, Biotechnology and Food Research Institute, Fukuoka Industrial Technology Center in 2001; Director, Technology Evaluation and Research Division, Ministry of Economy, Trade and Industry in 2005; Deputy Director, Evaluation Department, AIST in 2007; Director, Science and Technology Promotion Division, Nagasaki Prefectural Government in 2009; Director-General, AIST Chugoku in 2011; and Supervisory Innovation Coordinator, AIST from 2014. Doctor of Dentistry (Osaka University). In this paper, he was in charge of the structure of this paper and writing about the collaboration between regional centers and companies.

Yasufumi ESAKI

Graduated from Tsuru University in 1988. Joined a company formed as a result of merger of a major communication company and foreign company in 1988; and worked in sales at a city bank. Joined Sigma Corporation in 2002; Manager of President’s Office and Deputy Manager of Security Business Division; and currently, Chief Operating Officer, Sigma LIS Company. As the person-in-charge for promoting the development of mass production and business at the company side, has found important customers among automobile manufacturers and parts manufacturers, and was involved in the sales of over 200 inspection systems. In this paper, he was in charge of writing about needs and wants of companies, collaboration with AIST, and results of commercialization.

Discussions with Reviewers

1 Overall

Comment (Keiichi Ikegami, AIST)

This paper discusses the course of development and commercialization of a device system that conducts automatic and high-precision flaw and defect inspection of glossy and mirror surfaces, which are optically difficult to inspect, of inner walls of holes with various diameters. The “points of development” are clearly stated for each stage leading to the final commercialization, and the technical progress is described comprehensibly. In addition to technological hurdles, the ways in which unexpected hurdles such as the re-organization of the research institute and the Lehman Shock were surmounted are described, which makes this paper very thought-provoking.

Comment (Ken’ichi Fujii, AIST)

This research is very valuable on the point that automated inspection technology for glossy and mirror surfaces was developed, because flaws and defects were often missed in visual inspections of inner surfaces of holes with various diameters of industrial parts used in automobiles and others. This is a detailed report to show the process and the significance of using AIST's technological potential applying semiconductor laser diffraction, in order to develop an innovative defect inspection system, and to succeed in its commercialization. Particularly, this paper mentions that with overcoming crises such as re-organization of AIST and Lehman Shock, it was very effective to conduct product development matching demands of customers. This paper is an excellent paper that thoroughly discusses the scenario for successful product realization.

2 Demand for automated inspection technology

Question (Ken'ichi Fujii)

In Chapter 1, you write, "Visual inspection is done for the inner wall surfaces of machined holes with various diameters that are drilled in cylinder bores, automatic transmission valve bodies, hydraulic cylinders, and others that are important safety parts in automobile industry, since they have particularly strict inspection standards. As no oversight is tolerated, there is demand for high-precision and high-speed automated inspection technology that detects minute flaws and defects of about 0.1 mm." Please provide an easy-to-understand scale that shows the degree of effect of cost reduction through this automation, as well as the amount invested as cost to this type of inspection system by the automobile industry.

Answer (Yasufumi Esaki)

The cost of the system to realize automated inspection for valve bodies (transmission parts) is 15 million to 20 million yen/system. This system automates valve body hole inspection. In fact, about 60 % of the total items of inspection of valve bodies have become automated with this technology.

On the other hand, the normal cost of inspection personnel is about 10 million yen/person/year. Normally, hole inspection of valve bodies is conducted by four people/set, so automation will generate cost reduction of 40 million yen (10 million yen x 4) per year. Of course, improvement of quality was realized at the same time.

3 Development of defect inspection system for inner wall surfaces of machined holes

Question (Ken'ichi Fujii)

In Chapter 3, you write that you developed the technology for inspecting the inner wall surfaces of machined holes with excellent ideas such as installing optical fibers in the position in which specular reflection light can be received and the position in which only scattered light can be received, as shown in Fig. 8. What were the background and process that led to such ideas?

Answer (Saburo Okada)

When the authors were engaging in the research aiming to develop a noncontact shape measurement device as a new industrial measurement system utilizing characteristics of semiconductor lasers at the time, we got a request from a local steel sheet manufacturer on whether it was possible to realize a method and technology that could detect micro-defects of micron order on the surface of high-grade rolled steel sheets, and could also identify the difference between defects and roll marks. Therefore, we decided to develop an inspection system using laser beams. The issue at the beginning was the development of a sensor that allowed quick and efficient measurement of two-dimensional distribution of laser reflection light. At the time, it was difficult to obtain a special sensor that used concentric photodiodes developed in the USA. Instead, we devised an inexpensive sensor that we made ourselves. It was a multi-split planar detector made by bundling optical fibers, as shown in Fig. 6, and the objective was fulfilled.

During the same period, an automobile parts company requested the development of an inspection system for parts with mirror treatment on the interior of cylinders with a diameter of 25 mm and a depth of 150 mm. Using the reflection light image of Fig. 7 as a hint, we came up with a device shown in Fig. 8. Moreover, by increasing the focus depth by focusing the light gradually by long-focus lens after putting 200 mm or more of distance between the laser source and the flaw surface, and by optimizing the thickness, number, and tip position of the optical fibers, and after much trial-and-error, we were able to efficiently separate specular reflection light, scattered light, and diffracted light, and were able to greatly improve inspection performance.

Comment (Keiichi Ikegami)

The principle of this device has been mostly covered in Chapter 3, but I think the technological highlight of this paper is presented in Figs. 12 and 13. I think it will be more useful to the readers if you provide more detailed explanation featuring these figures.

Answer (Saburo Okada)

I added detailed explanations to Figs. 12 and 13.

Question (Keiichi Ikegami)

It seems that by reducing the diameter of the probe, the geometric condition for separating specular reflection light and diffracted (scattered) light became stricter. How did you work around this? If you could explain to the extent you are allowed, I think it will be easier to understand.

Answer (Yasufumi Esaki)

As a countermeasure against the reduction of data types, we made it possible to finely set the judgment conditions by adding algorithms. Every year, we improve the percentage of correct answers by adding 25 to 30 functions. At the same time, we also worked on the improvement of reception efficiency of laser irradiation and reflection light, to improve the quality of the obtained data.

4 Key to overcoming crises

Question (Ken'ichi Fujii)

You write that the collaborative research unit was established due to the re-organization of AIST in 2003, and that the potential demand survey was conducted during the recession period after the Lehman Shock to gain understanding of the potential demands such as for hole diameters of 20 nm or less and further time reduction, and these were greatly useful in the later product realization. You succeeded in downsizing the probe by developing a hollow motor as new transfer technology during the time, and this led to good sales performance after 2010 by increasing the degree of completion of the product. I imagine that there was much difficulty in starting new development during the economic recession. What were the way of thinking and policy that became the key?

Answer (Yasufumi Esaki)

During recession, the most important thing, I think, is how to prepare for the time when the economy improves. There was plenty of time due to slow business during recession, and we already knew the business potential of this inspection system due to our own surveys and projections. Therefore, I was thinking about how to push the business forward in the minimum time possible. First, we narrowed down the target, and set aim on the critical parts and important safety parts of automobiles. That is because we expected that the car companies would spend a lot of budget on automated inspection for parts subject to 100 % inspection in which there was no tolerance for oversight.

Next, since there was a limit to the manpower at the venture division of small and medium-sized companies, we conducted benchmark tests against competitive products, considered in which part we could differentiate ourselves in the parts inspection mentioned above, and set priorities to development topics that had high impact and high possibility of realization.

That is, we believed that if we narrowed down the target work that was most applicable to this inspection system, and if we could create a system that surpassed anything that the competitors had, we would be able to push the business forward when the economy improved.

Contributing to the SpaceWire international standard

—Successful factors for the development of a de jure standard—

Hiroki HIHARA[1]*, Masaharu NOMACHI[2] and Tadayuki TAKAHASHI[3]

[Translation from *Synthesiology*, Vol.11, No.3, p.148–158 (2018)]

Incorporating standards for spacecraft in Japan involves trading off various existing standards to comply with requirements and sustainability. However, well-established proprietary specifications developed for Japanese scientific satellites were successfully incorporated into the international standard of embedded networks, called SpaceWire, which was adopted for the X-ray astronomical satellite “ASTRO-H (Hitomi).” Looking back on this proposal process, we studied a mutual collaboration scheme to incorporate Japan’s proposal, regarding the development type international standards.

Keywords **:** De-jure standard, de-facto standard, SpaceWire, international standardization

1 Introduction

The onboard equipment installed in satellites is mutually connected by networks, and they transmit and receive commands and monitor signals called telemetry from each other. Standards for transmitting such signals are closely related to the ground stations that remotely control satellites and manage overall satellite systems. Since operating facilities of ground stations of various countries are used mutually, there is a growing demand for network communication standards to comply with international standards.

The process of introducing such international standards to Japan generally involves surveying several existing standards including for consumer products as well as spacecrafts, trading off with those standards, and selecting the specification that fulfills the required function and performance and for which continuity can be expected, after checking the background situation of standard establishment. On the other hand, in Japan, since proprietary development has been conducted for satellites used in scientific observations from the beginning, there are cases in which proprietary standards are being used.[1] For international standards, continuous revisions are conducted reflecting progress in technology, and this also involves revisions based on rapid technological advances in consumer product markets as well as establishment of new standards. While it should be possible to incorporate Japanese proprietary standards into international standards, this was not easy. This was not because of technological factors but because there seemed to be no motivation for proposing the Japanese proprietary standards as international standards for communication among the equipment onboard satellites.

The X-ray astronomy satellite “ASTRO-H (Hitomi)” dramatically improved functions and performances required for satellite systems compared with conventional Japanese satellites.[2]–[5] Development of equipment onboard ASTRO-H was conducted under wide-ranging international cooperation. Therefore, development that ensured continuation of the conventional development as well as compliance to international standard was required. The SpaceWire international standard[6][7] employed for ASTRO-H was a *de jure* standard that the European Space Agency (ESA) oversaw. We were able to incorporate proprietary standards that were formed with Japanese scientific satellites over the years into the international standard.

Looking back at the process by which Japanese proprietary standards were incorporated into the SpaceWire international standard and considering the success factors, it is possible to provide explanation based on the way of thinking presented in Reference [8]. The SpaceWire international standard was consolidated and established through discussions among the parties involved about the functions and performances to be realized, and its implementation method was set by specifying as international standards while technological development was conducted. This can be called a development-type standard.[9] In this article, we look back on the proposal activities based on the thoughts of Reference [8], and discuss the reproducible proposal process for the development-type international standard of which cases are increasing recently. Chapter 2 explains the route by which the system architecture of ASTRO-H, which is a compilation of SpaceWire standard products totally adopted for the first time, was recognized internationally. Chapter 3 summarizes the technological factors that allowed the Japanese proprietary technologies

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and proposals to be reflected in the international standard in the development process of ASTRO-H. Moreover, in looking back at the Japanese behavior pattern up to the moment the proprietary technologies and proposals were incorporated in the international standard, in Chapter 4, we compare this behavior with those of the American and European personnel referring to the model described in Reference [8] and consider the reproducible proposal process utilizing the Japanese behavior pattern.

2 System architecture of ASTRO-H

The development of ASTRO-H was conducted under international collaboration to realize an “open platform” described in Reference [10], as a situation Japan aims to realize in the future. This is a condition in which new ideas, technology, and people gather from around the world, and state-of-the-art added values are generated at a Japanese center of activity. It aims to make Japan the center for global intellectual activity. It also aims to create a structure to solve the problems that prevent the development of a spacecraft system that may produce innovative results, through cooperation transcending organizations. By lowering the threshold of joining the development of spacecraft systems, it provides opportunities for participation by a wide range of citizens.

As equipment installed in scientific satellites become diverse, the major issue is the difficulty of conducting development in a short time period while maintaining high reliability, as well as the complexity of the tests during the developmental process. Therefore, the R&D for spacecraft system architecture has advanced to conduct highly reliable design from the perspective of data handling and intercommunication among onboard equipment.[11][12] Scientific satellites involve a wide variety of mission purposes, such as near-earth or deep space observations. Since different forms have to be adopted, depending on their missions, one of the most important perspectives is to make a data handling system scalable for the architecture that can be used commonly in small and large satellites, rather than the concept of a fixed common bus. The network of electronic equipment installed onboard ASTRO-H that complies with the SpaceWire international standard was developed based on the “Future prospect of data handling system of scientific satellites” described in Reference [13]. The fully redundant[Note 1)] SpaceWire network that aimed for the SpaceWire international standard was realized for the first time in the world in ASTRO-H.[14] It was highly acclaimed in Europe where the standard was established, and ASTRO-H was introduced in the opening pages of the material published by ESA for the public, as shown in Fig. 1.[15]

3 Efforts by Japan

In developing the satellite onboard communication standard for ASTRO-H, approach was taken in which the personnel of Japan actively contacted the personnel of Europe and USA, conducted practical development and onboard demonstration from the planning stage of the standard. The team proposed improvement to the specifications that were derived through abundant achievement in Japan for the international standard. For the test and validation environment of the equipment that complied to the SpaceWire international standard, international joint R&D was done from the planning stage, and R&D and preparations were conducted to aim at unifying international understanding for handling the off-nominal[Note 2)] conditions that were not written in the specifications.[16]

It was the first time that a proposal from Japan was incorporated into the international standard for satellite onboard networks. The proposal from Japan was of a wide variety, from definitions of major protocol layers to fine correction of errors. In this chapter, we look back over the major three points that were incorporated into the standard, as well as the international joint R&D of the test and validation environment. These are culmination of

Fig. 1 SpaceWire international standard and ASTRO-H were presented in WELL CONNECTED, *European Space Agency Bulletin* (February 2011)[15]

experiences for the development of data handling systems such as scientific satellites, practical satellites, and space stations that have been developed by Japan.

3.1 Difference in viewpoint for optimal design

The Japanese proprietary specifications were utilized in the SpaceWire remote memory access protocol (SpaceWire RMAP), one of protocols in the SpaceWire international standard. RMAP is the protocol to read and write memories and others in the equipment connected to a network. Looking back at this process, it was found that there were two advantages to the Japanese development process.

One was the skill in obtaining consensus by smoothing communication among the organizations that carry out the R&D. In the communication standard layer that was initially being overseen by the SpaceWire Working Group Committee (SpW WG) that was set in the European Space Research and Technology Centre (ESTEC), a research institution of ESA, there was a layer added to realize real-time properties. It was called SpaceWire-RT or SpaceWire-T. In this proposal, the interface that directly linked to the uppermost telemetry command layer had a complex specification, and an agreement could not be reached for nearly a year because of heated discussions. From our experience of development and operation of the data handling system for Japanese scientific satellites, this protocol layer had a heavy implementation load and was not practical. Those of us participating in the SpW WG through the Japan Aerospace Exploration Agency / the Institute of Space and Astronautical Science (JAXA/ISAS) realized one point. Europe is a society that defines the content of one's job very precisely. The protocol layers of the communication standard had clear interfaces and could be easily divided into tasks, or enabled implementing specifications in parallel, and perhaps that was the reason there were overlaps in several portions in the layers. In contrast, the implementation the network protocol used in Japanese scientific satellites had the overlaps in each protocol layer skillfully removed. This indicates that the adjustment for each protocol layer was done adeptly under close communication among the parties involved when the specifications were compiled. We checked that RMAP itself was a protocol with sufficient function in providing real-time operation capability. Therefore, we pointed out that if we utilized this data format and communication protocol, SpaceWire-RT or SpaceWire-T were unnecessary in maintaining the real-time property, and imprinted our existing Japanese development specs in the form of an improvement proposal.[17] Moreover, Small Demonstration Satellite 1 (SDS-1) was launched in 2009, and the specification that we proposed was successfully demonstrated in orbit.

Figure 2 shows the communication standard layer that we proposed. We showed that what was initially done in eight layers or more could be done in seven layers, as shown in Fig. 2. The advantage of this communication standard layer is that it provides the real-time functionality required in onboard network for satellites by a simple protocol, based on the experience of development and operation of scientific satellites. The draft proposal was submitted at the 15th SpW WG in 2010 and obtained unanimous approval from the participating countries of ESA/ESTEC. This realized the simplification of the SpaceWire communication standard layers, and scalability could be realized from small satellites of 100 kg level to large satellites of 2.7 tons. We believe the simple and high-performance characteristic of SpaceWire could not have been obtained if this proposal was not submitted from Japan.

Another advantage is the point that we were able to respect the positions of the participants of all countries even in midst of the standardization proposal. As mentioned before, we swiftly launched SDS-1 in 2009 and succeeded in demonstrating the SpaceWire RMAP standard in orbit for the first time in the world. At this point, we felt for the first time that ESA trusted us. However, it was not because our technological level was demonstrated. They worried whether the standard on which they were working would be operational in orbit, or not. Instead of reporting that Japan was successful in onboard demonstration of the SpaceWire RMAP in orbit, we reported that the draft standard specification on which we were working that was the result of SpW WG was successfully demonstrated in orbit. In consequence, the results of the orbital demonstration by Japan wiped out their worries, and its success was shared by all parties involved. It seemed this led to the trusting relationship.

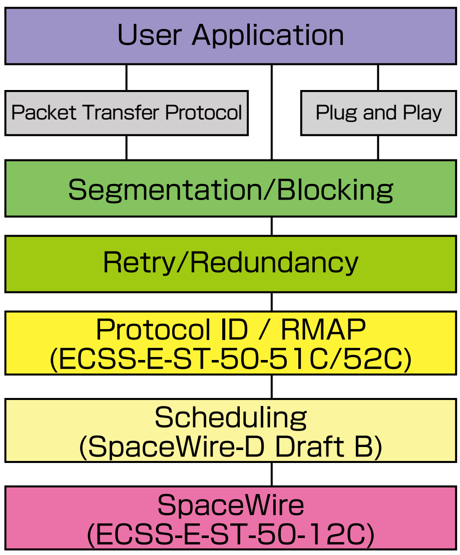

Fig. 2 Communication standard layer proposed to SpW WG from Japan[17]

3.2 Concurrent scheduling of time slot

The framework for maintaining sufficient real-time property on the SpaceWire network was greatly simplified based on the Japanese proposal mentioned earlier, and a design guideline was published as SpaceWire-D.[18] In the initial SpaceWire-D standard proposal, only one communication transaction per one time slot was allowed. This was based on the European claim that if multiple transactions were allowed in one time slot, the real-time property could not be verified, and the proposal was made as a standard of simple scheduling.

However, we have developed and operated a data handling system that enabled multiple transactions of communication within one time slot for many years, and had sufficient orbital experience. Although the practicality of this specification was empirical, JAXA knew the European culture that placed importance on formal and logical verification. Therefore, a government-academia-industry joint research plan was promoted, and through joint R&D by JAXA, Nagoya University, and the industries participating in the project, we created a guideline that could handle the European way of thinking in which importance was placed on the logical (formal) verification.[19] Based on this experience, we made proposals to implement multiple transactions of communication within one time slot, and this was reflected in the above specification as the concurrent scheduling of time slots. Here, the European claim that multiple communication transactions within one time slot could not be verified was reviewed, and specs were created as design guidelines to realize verifiable real-time performance. This was a result of the fusion of empirical knowledge that reflected the experience of the development and operation of the Japanese satellites, and the explicit knowledge of Europe that placed importance on logical integrity and verifiability.

3.3 Plug-and-play

The SpaceWire RMAP standard has several similarities to the specification of the peripheral interface module (PIM)[1] that had been conventionally used in Japanese scientific satellites as the communication standard. Based on the orbital operation experience in Japan, in ASTRO-H, the RMAP functions were utilized to define the address range that could be commonly referenced with the addressing mode called the standard RMAP address space that encompassed the common address space for the whole network. In this address range, when a certain address was accessed, the address and the communication service were linked so the data exchange (communication service) could be done with a communication protocol corresponding to that address. This feature was referenced at the SpW WG, and in the SpaceWire plug-and-play standard (current Network Discovery Protocol),[20] the specification was set so the standard RMAP address space set in ASTRO-H could be applied. As a result, the concept of “plug-and-play that links the satellite onboard equipment as if we plugged into an outlet” was realized.

Plug-and-play is a concept that is generally applied to consumer products, and in Japan, it was thought that the application to spacecraft onboard equipment was not very realistic. On the other hand, PIM that was Japan’s proprietary standard was similar to the plug-and-play concept defined by Europe, and this led to the actual specification proposal.

3.4 Results of Japan-Europe joint development

ASTRO-H was developed with the goal of connecting each equipment “like plugging them into an outlet” so they could be immediately tested or operated. Therefore, a test and validation environment was prepared considering unit tests, procurement plans, and subsystem tests, not only the development of equipment and subsystems. Moreover, expecting that the development would be conducted under wide-ranging international cooperation, the joint R&D for the RMAP conformance tester was conducted jointly with the University of Dundee that was overseeing the specifications for SpaceWire subcontracted by ESA.[16] This allowed the development of test specifications and pass-fail determination including responses under off-nominal conditions that are not clearly written in the specifications to be conducted in Japan. The devices that were developed in various countries were brought to Japan, and this allowed thorough development of a full redundancy network for large 2.7-ton satellites.

In the RMAP conformance tester, there were about 80 % of off-nominal test cases that were mutually understood and extracted in the process of the Japan-UK joint R&D. The off-nominal test conditions are not clearly written in the standard specification. However, careful investigation of off-nominal test conditions not only enables exact test and validation, but also detects insufficiencies and defects in the setting of nominal test conditions. As a result of such steady R&D, the RMAP standard which matched the understanding and requests of the personnel of Japan and UK (and Europe) was established. The RMAP conformance tester is used around the world as a *de facto* standard, and this includes both cases of nominal and off-nominal test. As a result, both the nominal and off-nominal conditions were guaranteed to have conformity and understanding, and if Japan purchased overseas equipment that complied with the SpaceWire international standard, they could be installed onboard the Japanese satellite system. For the tester for SpaceWire, joint development is being conducted with other European companies, and continuous cooperation is pursued to maintain conformity and international understanding.

4 Comparison of behavior patterns of each country

In the previous chapter, we summarized the technological elements in the Japanese proposals that were reflected in the SpaceWire international standard and reviewed the standard proposal activities. To extract the success factors that enabled the Japanese proposals to be reflected in the international standard, we shall look at the behavior patterns of the Japanese participants, not just the technological accomplishments. In this chapter, the basic loop for a specific subject to evolve continuously, as explained in Reference [8], will be used as a model for the discussion.

4.1 Reference model

The basic loop referenced in this article is shown in Fig. 3.[8] The blocks shown in the figure are autonomous entities that include humans (individuals, organizations, society), and there is no integrator that controls the whole. The condition of the subject is observed by the observer, and the observer sends out an alert as it interprets the meaning of change in status. The configurator thinks and gives advice on the action that should be taken when the alert is sounded. The actor voluntarily selects the advice, and acts based on such advice. The behavior assimilates with the subject and changes the condition of the subject. When the change is observed again, the information circles along the loop. As a result, the subject evolves. As it can be seen, interpretation, conception, selection, and assimilation are done autonomously rather than heteronomously, and this means that each block is a self-governing or autonomous entity, and this is thought to be the condition of evolution.[8]

In fitting the SpaceWire international standard to this basic loop for consideration, the condition is that each block is an autonomous entity. Specifically, SpW WG corresponds to the observer, and the European Cooperation for Space Standardization (ECSS) that is called the Technical Committee (TC) corresponds to the configurator. The vendors of the industrial world correspond to the actor, and the subject is the onboard satellite device or the communication standard for data that are exchanged among the equipment. Although the SpaceWire international standard is the *de jure* standard set by Europe, the SpW WG is placed in the preliminary stage of the TC that establishes the international standard, and it is accepted that the participants to this WG are autonomous beings. The participants can give individual opinions, and the vendors who are also actors can participate in the WG. They do not have to be representatives of national space agencies. This is different from the standard establishment process by country representatives that was the general practice for conventional communication standard establishment for spacecraft onboard equipment. It is a case of development-type standardization of which cases are increasing recently.[9] Moreover, the European vendors are allowed to participate in the ECSS which is the configurator. In the following sections, the Japanese behavioral pattern in the SpW WG is fit into the basic loop and is compared with European and American behavioral patterns. In this discussion, a member of each block may overlap, and the arrows represent the roles of how they approach each other.

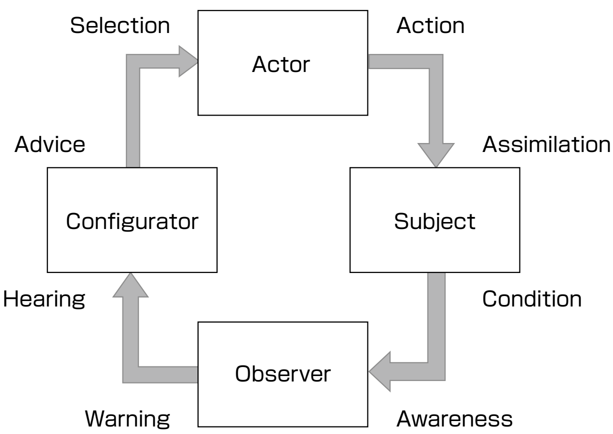

Fig. 3 Basic loop required for continuous evolution of a certain subject[8]

4.2 Behavioral pattern of European participants

The observer-configurator and the actor are separated and there is a division of labor. The observer-configurator is a governmental organization represented by ESA and may include system and equipment vendors. The actor is often a hardware or software vendor, and in some cases equipment development divisions of system vendors may be included. The division of labor between the former and latter groups is clearly separated in the specifications, and while there are frequent information exchanges such as conversations among the two groups, it is not common to see a case in which the work overlaps. That is, the work of investigating the specifications, and the work of manufacturing equipment to which the specification is applied almost never overlap. The actor waits for the specs and order from the observer-configurator, and the observer-configurator waits for the results of the actor to be reflected in the subject.

This behavior pattern will be explained by separating the observer's place and configurator's place, as well as the observer and the configurator in the basic loop shown in Fig. 3. A member participating in a certain place becomes clearly aware of the role allotted to the place, the source from which information needed for decision-making is obtained, and the place to which the contents of the discussions are to be transmitted. In establishing the SpaceWire standard, the observer's and configurator's places are designated, and research institutes, universities, and companies are able to participate in these places. For the observer's place, participation from outside Europe is not denied, and in some cases, such participation is encouraged. The observer and configurator may overlap, but when discussions are done in the observer's place, one must be aware that one's standpoint is about observation of the subject. When discussions are done in the configurator's place, the reports from the observer's place are received as formal reports, while the reports from the actor are not used directly for decision-making. The person who is an actor may participate in the SpW WG that is an observer's place and may give his opinions, but the actor does not engage in activities such as prototype making when it is in the observer's place. That is, it seems that the authority in establishing international standards is controlled by gathering and limiting the configurator's input to the observer's output. The aforementioned TC corresponds to the configurator's place, and only those selected within Europe can participate. Here, the observer's reports and standard proposals are screened. The observer's place has the authority to propose but does not have the authority to establish the standard. The authority to establish the standard is held by the TC or the configurator's place. This is shown in Fig. 4. In Fig. 4, the members are shown in rectangular boxes with sharp corners, while the places are shown as rectangles with rounded corners.

The reason why the observer's and configurator's places are separated is because the standard proposals are compiled after conducting adjustments within Europe. Manufacturing and specification settings are structurally separated, and the participants of the observer's and configurator's places have low awareness of being the actors. For example, in cases in which personnel from industrial vendors take roles of the observer or the configurator, it often happens that they must move to other organizations.

4.3 Behavioral pattern of participants from USA

There is no clear hierarchical awareness for the observer, the configurator, and the actor among the participants from the USA. The observer acts on the subject as the actor and expects quick feedback. Also, they expect the SpW WG, which was originally set up to be in the observer's place, to have the mindset of the configurator, and to directly make standard proposals. That is, they are not aware of the limitations of the observer's place. While the authority and the role of the TC as being in the configurator's place are recognized, they think it is possible for the actor to directly propose in establishing standards at the configurator's place. They propose specs gathered through the actor's performance in the market to be used as international standard specifications, and therefore, expect discussions on considering them as *de facto* standards. This means that the information between the actor and the configurator flows in both directions. This is shown in Fig. 5. The bidirectional arrow between the actor and the configurator, as shown in this figure, does not match the behavior pattern of the European participants in Fig. 4. That is, this model shows that the behavior pattern of American participants is not accepted in Europe.

While the hierarchy of the observer, the configurator, and the actor not being separated is similar to the Japanese behavior pattern, it is not uncommon that the observer and the configurator share common interests as actors. In such cases, the Japanese participants tend not to be able to compete, but the Europeans consider the WG Committee as the observer's place, and here, the proposal of specs based on existing performances as international standards or as the *de facto* standard is not accepted. For the Japanese participants, it can be thought that this is an opportunity given to objectively state their opinion.

4.4 Behavior pattern of Japanese participants

Japan has a setup of establishing Japanese standards through the supervision of JAXA. In the establishment of the SpaceWire standard, there is no clear division of labor as in the European example of SpW WG that corresponds to the observer's place or the ECSS that corresponds to the configurator's place. Instead, a design standard working group is set up as the configurator's place for standard establishment. The configurator's place has a high degree of independence, and there is no structure in which the observer definitively acts on the configurator as seen in the European example. Members from the national research institutions,

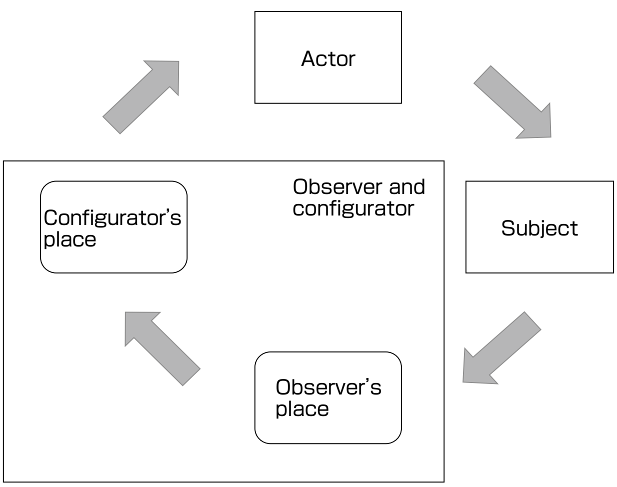

Fig. 4 Behavior pattern of European participants

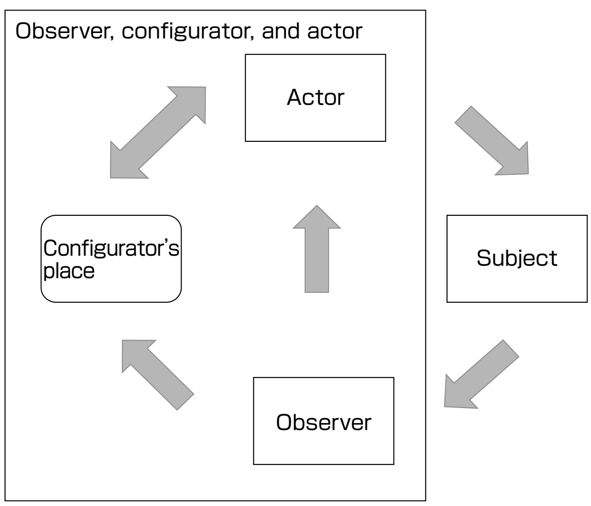

Fig. 5 Behavior pattern of American participants

universities, and companies participate in the design standard WG that is a configurator's place to establish the standard. The members in universities and research institutes often act as observers in the standard establishment process, and may become actors during the R&D. Also, the manufacturing companies that normally act as actors may become observers as system vendors. This is thought to be due to the background that the Japanese space development has been conducted jointly by government and private companies. In this case, it is possible to incorporate output from an overseas configurator's place, and it is also possible to quickly realize international standards in products. This is shown in Fig. 6(a). It is not uncommon that the observer places an order to the vendor who is the actor based on proprietary specifications, without consulting the configurator. In such cases, the subcontract specifications correspond to the arrow that points from the observer to the actor, but the content of subcontract specifications in many cases is based on the mutual interaction between the actor and the observer, and the arrow should go in both directions. Only after accumulation of experiences, the standard is established formally at the configurator's place, and its promotion is done by the actor. The configuration shown in Fig. 6(a) has no arrows of influence that conflicts with the European behavior pattern and can be superimposed well. This is shown in Fig. 6(b).

The SpW WG, as mentioned above, accepts participants of those who stayed outside Europe to the observer's place, and has the mechanism of unitarily incorporating the output of the observers' place into the configurators' place. The Japanese participants participated in the observer's place with the consciousness of both the observer and the actor. This consciousness did not cause conflict in the Working Group Committees. This was in contrast with the conflict between the American and European participants generated by the American way of thinking of bidirectional awareness in the observer's and configurator's places.

The participants from Japan had a unified way of thinking of the actor and the observer, and the example of SDS-1 mentioned earlier shows that the Japanese behavior pattern shown in Fig. 6 fits well with the European behavior pattern shown in Fig. 4. The results of the American preceding development could be brought into the configurator's place without stress through the Japanese proposals, and it is thought that this contributed to bridging the gap between the USA and Europe.

On the other hand, issues Japan faced became apparent. For the technological investigations discussed in the SpW WG, there were not any apparent differences in terms of the technology level between USA's National Aeronautics and Space Administration (NASA), Europe's ESA, and Japan. However, Europe and USA had many orbital demonstrations of new technologies and plenty of examples of new equipment being used. What is the cause of this difference? The participants of the SpW WG included about 14 countries from Europe, USA, the Far East, and Asia. The countries engaged in their original development, and some are leading in demonstrations of new technology. The performances were referenced to determine specifications to be aimed at. Risks of practical utilization were indicated from various perspectives. Even if the problem was pointed out, alternative plans were proposed actively. The claims and proposals of Japan were adopted without discrimination if they were reasonable, backed by experience, and matched the direction of SpW WG. The fact that there was a place for gathering

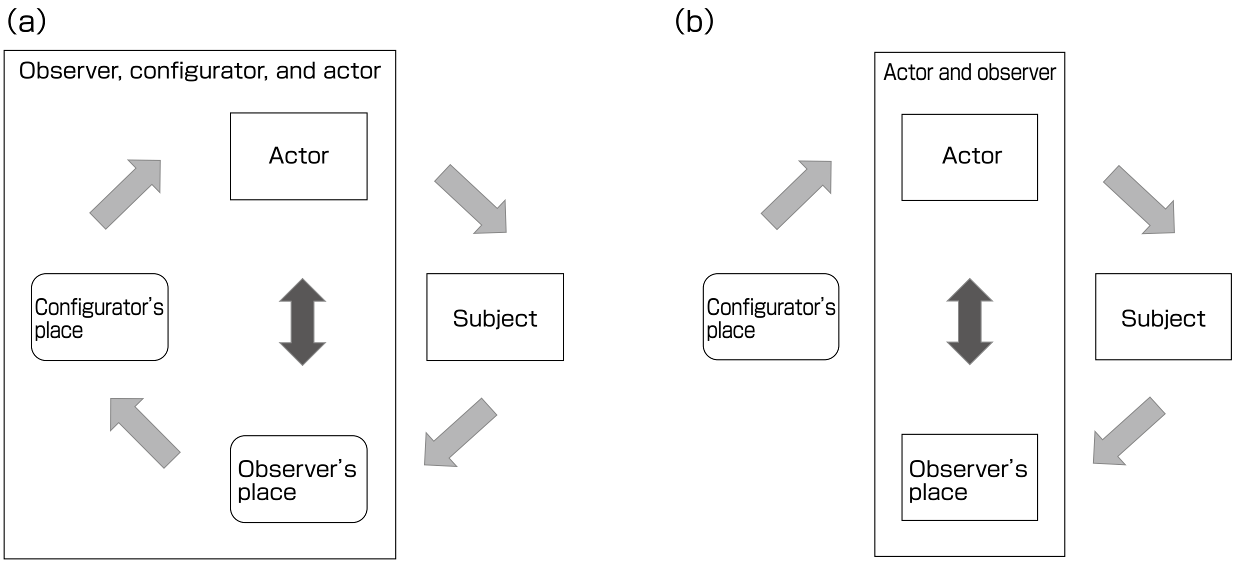

Fig. 6 Behavior pattern of Japanese participants
(a) Domestic standard establishment process; (b) combination with European *de jure* standard establishment process.

and listening to specialists with various backgrounds led to innovation, cost reduction, downsizing, and weight reduction, and as a result, linked to the "precedents of Europe and USA." That is, there is a need for a platform to be prepared to gather skills and knowledge of the participants who have diverse backgrounds toward a common goal. The issue was to prepare such a platform in Japan.

5 Summary

In this article, we reviewed the process by which the Japanese proposals were reflected in the SpaceWire international standard, and the success factors were considered referring to the conceptual model for continuous evolution of the subject described in Reference [8]. In looking back, we were able to reconfirm that the Japanese proprietary technologies were valid internationally, and the Japanese behavior pattern of respecting the preceding technology while conducting "kaizen" was effective in incorporating (the word "rub in" perhaps better describes the situation) the proprietary technology into international standards. As a result, we believe we were able to describe the empirical knowledge for reflecting the Japanese proposal in an international standard utilizing the European *de jure* standard establishment process as a reproducible model. By being aware of this model, we hope the activities for getting the Japanese proposals reflected in international standards will be activated.

6 Acknowledgement

For the practical realization of SpaceWire, we are grateful to Dr. Takayuki Yuasa (Spire Global, Inc.) who was deeply involved in the development of the SpaceWire network for ASTRO-H at ISAS. We thank Shimafuji Electric Inc. that developed test equipment that swiftly incorporated the SpaceWire international standard specs and contributed to its practical realization. The discussions in this article were inspired by the lectures on advanced regulatory science by Professor Yasunori Baba, the Research Center for Advanced Science and Technology, The University of Tokyo. He provided us with a hint that it might be possible to construct a reproducible model. We are thankful to Professor Baba who made us aware that it is possible to link the success stories of standardization activity, which tend to end up as stories of personal experiences, to practical application.

Note 1) All onboard units have redundant systems.
Note 2) It is not the normal condition that is set in the specifications.

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Discussions with Reviewers

1 Overall

Comment (Akira Ono and Motoyuki Akamatsu, AIST)

For the international standardization of SpaceWire that is the communication standard for onboard satellite networks, the scenario by which the Japanese proposal was adopted is described from the technological background and the roles of people involved. The Yoshikawa model was applied for the development-type standard establishment, and analysis is done for the behavior patterns of European, American, and Japanese practitioners of standard establishment. This process can generally be applied to any standard development, not limited to the satellite communication standard, and therefore this article is appropriate for publication in *Synthesiology.*

2 Range and role of the participants in standard establishment

Question (Akira Ono)

So-called international standards include ISO and IEC, and the international standards are created by agreement among the standardization institutions of various countries. For the SpaceWire international standard, the main subject of this article, what kind of people and organizations were involved and agreed to create this international standard? I imagine that the space technology specialists from around the world, space agencies of various countries, ESA, and others were involved, but which entities played what roles in creating the SpaceWire international standard?

Answer (Hiroki Hihara)

SpaceWire was originally proposed by the European Space Agency/European Space Research and Technology Centre (ESA/ESTEC). It started this investigation with the objective of creating a standard for inter-device communication onboard spacecraft without using special parts for military use that were conventionally used in spacecraft. International standards are established within ESA by the Technical Committee (TC) that is part of ESTEC. However, prior to the establishing process at the TC, the SpaceWire Working Group Committee (SpW WG) was set up to take the role of discussing specifications and then submitting the specs to the TC.

There is no restriction to the qualifications to participate in the SpW WG. People of any country, regardless of whether they belong to government, academia, or industry can participate.

There is no restriction to the right to speak in the SpW WG, and anyone can speak freely. In fact, space agencies, universities, and companies of Europe, Japan, Russia, and USA are participating, and space-related government organizations and research institutes of Turkey, Brazil, and others participate occasionally.

However, I do feel that there is a tacit understanding about whom Europe will accept as participants. That is, although there is no clear qualification, only those who are capable of actually conducting spacecraft R&D and are able to propose and discuss specifications are accepted as participants.

3 Development-type standard

Question (Akira Ono)

You use the terminology "development-type standard," but how is this different from ordinary standards, and what is its definition? Please explain why the SpaceWire international standard is a "development type." Also, if something is a development-type international standard, what is the author's thoughts on the points that must be considered in such standardization compared to conventional standards?

Answer (Hiroki Hihara)

The terminology, "development-type standard," is taken from Reference [9]. It was cited from Masami Tanaka's *Kokusai Hyojun No Kangaekata—Global Jidai He No Atarashii Shishin* (Dialogues on International Standards—A Guide to the Global Age) (University of Tokyo Press, 2017). I understood the terminology, "development type," as a situation where there is a preceding objective for development, and the discussions begin from the standard system that is necessary for achieving the objectives and the types of standards. Organizations that participate in the SpW WG are expected to present the results of their R&D, prototype evaluation, and orbital demonstrations which they conducted. Moreover, all presentations are respected. I think those situations can be expressed by the term, "development type."

In conducting the standardization proposal, although the SpaceWire itself is a communication standard, there were recommendations for connectors and semiconductor devices, there was tolerance for introducing new technology while guaranteeing the reliability required for spacecraft, and the SpW WG participants were expected to pursue advanced functionality and performances. I felt this led to tacit understanding, and this is the point to which one must pay attention.

Also, there was reorganization of the range to which the standard applied, and the scheduling of standard establishment was unclear. The companies must be able to continue product development and make proposals by actively disclosing the parts where compatibility with other organizations was necessary in their product specs. The ability to make proposals even under an uncertain schedule is another point to note.

4 Organizations that correspond to observer, configurator, and actor

Question (Akira Ono)

Please explain which organizations correspond to the "observer," "configurator," and "actor" that you mention in "Chapter 4 Comparison of behavior patterns of each country" when the case is applied to Japan. Is my understanding that follows correct: "subject" = satellite or onboard sensor and/or data and information obtained from them; "observer" = research institution, university, researchers of JAXA, and/or data users; "configurator" = JAXA; and "actor" = manufacturing companies? Does such corresponding relationship apply to overseas organizations?

Normally, a standard is considered to be an agreement to which product providers and users are expected to comply in carrying out commercial trade, but in this article, to which entities do the product providers and users correspond?

Answer (Hiroki Hihara)

In Japan, there is a mechanism for domestic standard establishment that is overseen by JAXA, and the standard is established based on the discussions between the manufacturing companies which are the actors and JAXA which is the observer, concerning the satellite and observation data that are the subject.

The universities and national research institutions often take the standpoint of observers in the standard establishment process, and take the standpoint of actors in R&D. Also, the manufacturing companies with the characteristic of system vendors may participate in standard establishment with the perspective of observers. Since standard establishment is done by the standardization committee consisting of participants that share a neutral position that keeps them independent from their respective organizations, the configurator's place has high independence, and I think this is related to the background that the technological development of spacecraft in Japan was conducted through cooperation between public and private sectors.

In cases of overseas countries, Europe does not interfere with the research institutions which they are not space agencies, as long as it is for the SpaceWire standard. I do have an impression that there is a clear division of role among the research institutions according to their standing. In the USA, universities are not involved in standard establishment, and only NASA and the companies are involved. Therefore, both Europe and USA have different response to the reference model compared to Japan.

In the model referenced in the article, I think the aforementioned difference can be expressed by referring to the provider and the user as the actor and the observer. However, in the case of Europe, there is a way of thinking that the standard can be used as part of the structure of commercial trade, and I think the observer can take the standpoint of the configurator and become the provider of the structure of trade.

5 Cause of difference of behavior pattern of each country

Question (Akira Ono)

In Chapter 4, you explain the differences of the behavior patterns of Europe, USA, and Japan. What do you think is the main reason there are different behavior patterns among the countries?

Answer (Hiroki Hihara)

Through experiencing the process of SpaceWire standard establishment, I think the main reason that generates the difference in behavior pattern is the difference of policy for nurturing industry. Europe aims for coexistence that does not favor elimination and avoids the risk of stagnating progress by reaching an agreement through discussion that allows the presence of different values. USA consciously accepts elimination and promotes progress through selection of proposals. Japan seems to position standards as mediation means rather than a way for nurturing industry.

6 Issues for Japan

Question (Akira Ono)

Compared to Europe and USA, what do you think is the issue for Japan in terms of behavior pattern?

Answer (Hiroki Hihara)

Compared to Europe and USA, I feel that there is no place to make use of diversity in Japan. In Europe, common sense based on tacit understanding seems to exist in each European country, and there is a place for discussion while respecting the differences in values and experiences of different countries, and this is useful in explicitly utilizing diversity.

USA has diversity within itself, and it is conscious that achievement can be attained through different viewpoints and is clearly aware that diversity is the source of their strength. Japan has diversity within its country, but I feel there is no place to share the achievements that arise through diversity.

Question (Motoyuki Akamatsu)

You explain that it was not easy to incorporate the Japanese proprietary standard into the international standard, but please explain why it was not easy.

Answer (Hiroki Hihara)

On the point that you indicated, I think the main reason was motivation, and I added this in the text.

7 Joint research with universities and standardization

Question (Motoyuki Akamatsu)

You write about the concurrent schedule, and I understood that the point here is that you constructed the design guideline through joint research in addition to the orbital demonstration. Was this joint research part of the scenario that aimed to take this to international standardization? If so, based on what decision was this joint research conducted?

Answer (Hiroki Hihara)

Looking back over the joint research, I think Professor Takahashi, who was the project manager of “Hitomi” project, was knowledgeable about the European culture that placed emphasis on formal and logical verification. I am now aware that he worked on the government-industry-academia joint research plan to prepare the specifications based on empirical knowledge into a form that was acceptable in Europe, and in this joint R&D, he constructed the guideline that made possible the scrutiny of European way of thinking that placed importance on logical (formal) verification. I added this point to the text.

8 Future contribution

Question (Akira Ono)

This article describes a case in which Japan contributed greatly to the creation of an international standard for space technology. What is the most important point if Japan wishes to continue such contribution in the future? It can be on general technology other than space technology.

Answer (Hiroki Hihara)

Looking back at the process of SpaceWire standard establishment, I think the most important thing in the future is the will to work not only on preceding examples but also on diversity. There are two reasons for this as follows.

First, with the thought of so-called “kaizen,” rather than the mind of competition that starts by negating the existing results, it was found that we could contribute to the establishment of international standard by respecting the existing results. This originates from the fact that the PDCA cycle can be turned quickly because we have a culture of unity where there is no hierarchy between the side that establishes the specs and the side that uses the specs.

Next, the kaizen method starts from the fact that there is a preceding case. With the improvement of the technological level in Japan, it has become difficult to find a precedent. In the future, we can keep the preceding cases in view by eliminating the idea of advanced versus developing countries, or new versus old technologies, and changing the awareness to capturing the precedents through expression of diversity.

From these reasons, diversity should be considered as precedents, should be respected, and should have kaizen applied. Then we can coexist with the values of competition of Europe and USA and shall be able to continuously contribute to international standard establishment.

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 April 1, 2017

1 Types of articles submitted and their explanations

The articles of *Synthesiology* include the following types:
• Research papers, reports, commentaries, roundtable talks, and readers' forums
Of these, the submitted manuscripts of research papers, reports, and commentaries undergo review processes before publication. The roundtable talks are organized, prepared, and published by the Editorial Board. The readers' forums carry writings submitted by the readers, and the articles are published after the Editorial Board reviews and approves. All articles must be written so they can be readily understood by the readers from diverse research fields and technological backgrounds. The explanations of the article types are as follows.

① Research papers
A research paper rationally describes the concept and the design of R&D (this is called the scenario), whose objective is to utilize the research results in society, as well as the processes and the research results, based on the author's experiences and analyses of the R&D that was actually conducted. Although the paper requires the author's originality for its scenario and the selection and integration of elemental technologies, whether the research result has been (or is being) already implemented in society at that time is not a requirement for the submission. The submitted manuscript is reviewed by several reviewers, and the reviewers will recommend whether the manuscript should be accepted, revised, or declined. The author completes the final draft based on the discussions with the reviewers. Views may be exchanged between the reviewers and authors through direct contact (including telephone conversations, e-mails, and others), if the Editorial Board considers such exchange necessary.

② Reports
A report describes a development example of technology which has practical value as well as an example of new technology which has been put to practical use. It contains 1) the aim, 2) the process of development (the course to the goal), and 3) the outcomes. The submitted manuscript is checked by the Editorial Board. The authors will be contacted if corrections or revisions are necessary, and the authors complete the final draft based on the Board members' comments.

③ Commentaries
Commentaries describe the thoughts, statements, or trends and analyses on how to utilize or spread the results of R&D to society. Although the originality of the statements is not required, the commentaries should not be the same or similar to any articles published in the past. The submitted manuscripts will be checked by the Editorial Board. The authors will be contacted if corrections or revisions are necessary, and the authors complete the final draft based on the Board members' comments.

④ Roundtable talks
Roundtable talks are articles of the discussions or interviews that are organized by the Editorial Board. The manuscripts are written from the transcripts of statements and discussions of the roundtable participants. Supplementary comments may be added after the roundtable talks, if necessary.

⑤ Readers' forums
The readers' forums include the readers' comments or thoughts on the articles published in *Synthesiology*, or articles containing information useful to the readers in line with the intent of the journal. The forum articles may be in free format, with 1,200 Japanese characters or less. The Editorial Board will decide whether the articles will be published.

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*, except 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

3.1.1 Articles may be submitted in Japanese or English.
Accepted articles will be published in *Synthesiology* (ISSN 1882-6229) in the language they were submitted. 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 within 2 months after the original edition is published.
3.1.2 Research papers should comply with the structure and format stated below, and reports and commentaries should also comply with the same structure and format except subtitles and abstracts are unnecessary.
3.1.3 Research papers should only be original papers (new literary work).
3.1.4 Research papers should comply with various guidelines of research ethics

3.2 Structure

3.2.1 The manuscript should include a title (including subtitle), abstract, the name(s) of author(s), institution/contact, main text, and keywords (about 5 words).
3.2.2 Title, abstract, name of author(s), keywords, and institution/

contact shall be provided in Japanese and English.

3.2.3 The manuscript shall be prepared using word processors or similar devices, and printed on A4-size portrait (vertical) sheets of paper. The length of the manuscript shall be, about 6 printed pages including figures, tables, and photographs.

3.2.4 Research papers, reports, and commentaries shall have front covers and the category of the articles (research paper, report, or commentary) shall be stated clearly on the cover sheets.

3.2.5 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.

3.2.6 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 society.

3.2.7 The abstract should be 300 Japanese characters or less (125 English words). The Japanese abstract may be omitted in the English edition.

3.2.8 The main text should be about 9,000 Japanese characters (3,400 English words).

3.2.9 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 contributions to the paper are included.

3.2.10 Discussion with reviewers regarding the research paper content shall be done openly, 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 with the names of the reviewers disclosed. The edited discussion will be attached to the main body of the paper as part of the article. Regarding the reports and the commentaries, discussion with the Editorial Board members will be opened at the Board's discretion. In this case, the Editorial Board will edit the discussion to about 800 Japanese characters (less than half a page) with the names of the Board members disclosed.

3.2.11 If there are reprinted figures, graphs or citations from other papers, prior permission for citation must be obtained and should be clearly stated in the paper, and the sources should be listed in the reference list. A copy of the permission should be sent to the Publishing Secretariat. All verbatim quotations should be placed in quotation marks or marked clearly within the paper.

3.3 Format

3.3.1 The headings for chapters should be 1, 2, 3…, for subchapters, 1.1, 1.2, 1.3…, for sections, 1.1.1, 1.1.2, 1.1.3, for subsections, 1.1.1.1, 1.1.1.2, 1.1.1.3.

3.3.2 The chapters, subchapters, and sections should be enumerated. There should be one line space before each paragraph.

3.3.3 Figures, tables, and photographs should be enumerated. They should each have a title and an explanation (about 20–40 Japanese characters or 10–20 English words), and their positions in the text should be clearly indicated.

3.3.4 For figures, image files (resolution 350 dpi or higher) should be submitted. In principle, the final print will be in black and white.

3.3.5 For photographs, image files (resolution 350 dpi or higher) should be submitted. In principle, the final print will be in black and white.

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

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

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

Website—[No.] Author(s) name (updating year): Title of web page, Name of website (may be omitted If the name of the website is the same as that of the author(s)), URL, Access date.

4 Submission

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

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c/o Public Relations Information Office, Planning Headquarters, National Institute of Advanced Industrial Science and Technology(AIST)
Tsukuba Central 1, 1-1-1 Umezono, Tsukuba 305-8560
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The submitted article will not be returned.

5 Proofreading

Proofreading by author(s) of articles after typesetting is complete will be done once. In principle, only correction of printing errors is 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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Letter from the editor

There are three research papers and one commentary in this issue. Both the Fukaya paper and Kimura paper are about porous materials. The former is about the technology to manufacture silica materials, which are already being widely used, from incineration residues, while the latter is about the synthesis technology to enhance the possibility of non-silica materials, aiming to achieve highly functional porous materials. On one hand, there is R&D conducted to realize an ecosystem in which high quality porous materials can be made from garbage incineration residues as energy saving technology of manufacturing silicon chemical products. On the other hand, there is concentrated research to pursue the possibility of porous materials. It seems that these are two extremes in the direction of research. Both are impactful R&D, and I believe they offer useful hints to those who are trying to figure out how to proceed with their research skills and technologies. The Okada paper is about the development and commercialization of a laser inspection system for the inner walls of machinery components, and it leads up to practical application of new technology while taking in various ideas through collaboration with a company, against a background in which there is strong demand for improvement of inspection technology as automation in manufacturing progresses. In *Synthesiology* Volume 11 Issue 1, the Furukawa paper also discusses the technological development of an inspection device that measures the precision of cylinders using optical measurement. It can be seen that the common points of the two papers are the collaboration with companies, and the major forces of collaboration are fusion of corporate technology, understanding of potential market demands, and passion to realize new technology. The Hihara commentary is about international standardization of communication standards of satellites called SpaceWire, and it is a description of the course by which the work done in Japan was adopted as the international standard. Here, the superiority and experience of communication technology developed in Japan were evaluated properly, and the paper also describes the behavior patterns of the people of Japan, Europe, and USA. Many readers who have been involved in international standardization activities may have similar impressions about the behavior pattern, and I think it may serve as a guideline for how to conduct discussions on the international stage.

(Motoyuki AKAMATSU, Executive Editor)

Aim of *Synthesiology*

— Utilizing the fruits of research for social prosperity —

There is a wide gap between scientific achievement and its utilization by society. The history of modern science is replete with results that have taken life-times to reach fruition. This disparity has been called the *valley of death*, or the *nightmare stage*. Bridging this difference requires scientists and engineers who understand the potential value to society of their achievements. Despite many previous attempts, a systematic dissemination of the links between scientific achievement and social wealth has not yet been realized.

The unique aim of the journal *Synthesiology* is its focus on the utilization of knowledge for the creation of social wealth, as distinct from the accumulated facts on which that wealth is engendered. Each published paper identifies and integrates component technologies that create value to society. The methods employed and the steps taken toward implementation are also presented.

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Highlights of the Papers in *Synthesiology*

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