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Submarine earthquake- and tsunami-induced event deposits

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Materials recycling technology for recovering rare earth fluorescent powder from fluorescent lamp sludge

Synthesiology Editorial Board



2018



# 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

# Development of low sticky texture natto "MAMENOKA"

# Yuji Киво et al.

This paper describes the effort to promote natto, a product of a major local industry of Ibaraki Prefecture, in foreign countries. Breaking away from the customary ways of consuming natto in Japan, many companies were able to achieve product realization, through diverse collaboration of industry-academia-government under a clear scenario, and by combining biotechnology, marketing, and cooking skills.

# Submarine earthquake- and tsunami-induced event deposits

# *—Disturbance of the sea floor by huge earthquakes and their related tsunamis, and the use of disturbance records in marine sediments for the history of past huge earthquakes and tsunamis—*

# Ken Ikehara et al.

Keeping in mind contribution to safe and secure living, this paper discusses the strategy to clarify the history of past giant earthquakes based on the geological evidences of seafloor deposits left after earthquakes and tsunamis. For earthquake and tsunami disaster prevention, not only are state-of-the-art surveys and analyses necessary, but also continuous data collection during normal times is important.

# High-accuracy endoscopic microscopy using a thin, 1.5 mm diameter probe with optical coherence tomography Hiromitsu Furukawa *et al.*

By combining the world's most advanced micromotor technology of a private company and the correction technology for optical coherence tomography (OCT) developed by AIST, an endoscopic microscope with diameter of 1.5 mm that allows the interior inspection of a cylinder at 20 nm accuracy was created. OCT was originally developed as medical technology, and this technology was applied to industrial use that involved completely different required performance.

# Materials recycling technology for recovering rare earth fluorescent powder from fluorescent lamp sludge *—Pioneering near-future resource circulation—*

# Tatsuya Oki et al.

In modern society, recycling is one of the most important topics. This paper discusses the practical realization of recycling technology for rare earths in fluorescent powder material, without reducing to the element level. It is a success story of the integration of advanced knowledge through the collaboration of researchers specializing in materials and those specializing in sorting devices.

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# **Development of low sticky texture natto "MAMENOKA"**

Yuji Kubo and Rikio Nakagawa\*

[Translation from Synthesiology, Vol.11, No.1, p.1–11 (2018)]

Many foreigners appreciate Japanese foods because of their health properties. However, few foreigners like natto, because of its sticky texture. The Ibaraki prefectural government started the "development of low sticky texture natto" project to export natto-based products to France and Germany, among other countries. The use of specially shaped packages, or edible glue lowers the stickiness of natto. However, these methods require new processing machines. Natto makers would like to produce low sticky texture natto without having to purchase new machines. To meet this challenge, we developed a new strain of bacteria from a natural mutation during subculture of Bacillus subtilis. This strain produces substances with less stickiness. We named this strain "IBARAKI <code>lst-1</code>" and called natto products fermented by IBARAKI <code>lst-1</code> "MAMENOKA." MAMENOKA means fragrance of fermented soybeans. We found that the stickiness and viscosity of MAMENOKA were significantly lower than that of commonly sold natto. Firmness, color, and nutrient (polyamine) content of MAMENOKA were almost the same as commonly sold natto. We performed taste tests of MAMENOKA and commonly sold natto. The majority of the people who tested MAMENOKA reported that it was easier to swallow than commonly sold natto, because of its low viscosity. Some natto makers in Ibaraki are utilizing these properties of MAMENOKA to develop new natto-based products, such as dipping sauces, pastes, and dressings.

Keywords : Natto, Bacillus subtilis, low sticky texture, IBARAKI & st-1, MAMENOKA

# **1** Introduction

The domestic consumption of natto has leveled off due to the effect of reduced rice consumption, and the overheating price competition is causing pain to the small and medium-sized companies. Then, *washoku* or Japanese food was registered as a UNESCO Intangible Cultural Heritage in December 2013, and interest in Japanese food has increased worldwide.

However, the diffusion of natto overseas has not reached far, except in Asia, and there is plenty of room for opening foreign markets.

Against such a background, the development of natto for overseas market started in 2013 through the efforts of the "Ibaraki Council for the Promotion of Growing Industry" promoted by the Industrial Policy Division, Ibaraki prefectural government. The coordinator of the Council thought that the sticky texture of natto was a negative factor in promoting this product overseas where there was no culture of using chopsticks or practice of slurping food, and it was proposed that the stickiness of natto should be reduced. The idea was that natto with low stickiness might be readily accepted overseas.

It was assumed that rather than eating natto as it is, it would be promoted as an additive food ingredient for preparing menus. The characteristic of natto that it has "high percentage of protein and low fat in comparison to the calories" was expected to be the appeal point for healthconscious consumers. The term "natto" generally refers to "sticky" or "stringy" natto. Therefore, in this paper, the sticky natto will be simply called "natto."

# 2 Objective and the scenario to achieve the objective

The objectives were to develop natto with low stickiness with the aim of selling natto as a new food ingredient overseas, and to diffuse the research results to natto manufacturing companies in Ibaraki Prefecture. The scenario to achieve these objectives was set in the order from (1) to (5) as follows:

- To clarify the necessary requirements to spread natto overseas
- (2) To develop and breed natto bacteria needed for overseas deployment
- (3) To conduct evaluation of characteristics of natto made from the newly bred natto bacteria
- (4) To understand the characteristics of the newly developed natto and to publicize this product to related people
- (5) To conduct activities needed for overseas diffusion including creation of menus for foreigners and participation in overseas food exhibitions

For (1), after discussions with the related parties, the requirements were set as development and branding of natto with low stickiness and proposal of natto menus that would be accepted by foreigners. For (2), literature search was conducted at the Industrial Technology Institute of Ibaraki Prefecture (Ibaraki ITI), and it was decided that selection and culture of natural mutant strains that are generated in the process of subculturing would be done.

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For (3) to (4), it was decided that the stickiness, hardness, color, and nutrients of natto would be evaluated, and sensory evaluation would be conducted. For (5), through discussions with related parties as in (1), it was decided that a brand name and logo would be selected, creation of new menus using natto would be contracted to a western cuisine specialist, and participation in worldwide food exhibitions held in Europe would be promoted and so forth. (Fig. 1).

# 3 Issues that had to be solved

There are several methods proposed for the manufacture of natto with low stickiness, and the following have been publicized as patent information<sup>[1]–[3]</sup>:

- Method by which the form of container is devised and natto is made by fermenting the soybean in the container
- (2) Method of adding a glue ingredient in the natto manufacturing process
- (3) Method of mixing cinnamon bark, cocoa, sugar, mayonnaise, and others to natto that uses okara (soy pulp) as its main ingredient.

The method for manufacturing natto with low stickiness

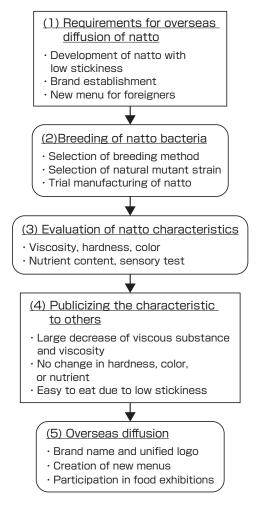


Fig. 1 Scenario for developing natto with low stickiness and its diffusion overseas

# Table 1. Conventional method to manufacture natto with low stickiness and method used by Ibaraki ITI

	(1) Working on container	
Conventional method	(2) Addition of glue	
	(3) Use of okara as raw ingredient	
Method used by Ibaraki ITI	Use of natto bacteria that was bred by repeated selection of natural mutant strain that arose during subculturing of natto bacteria	

by working on the container<sup>[1]</sup> involved a totally different manufacturing method compared to regular natto production, and it was difficult to utilize the already existing facilities of the natto companies. Therefore, facility investment cost was an issue. In the method in which a glue is added,<sup>[2]</sup> there was a problem of needing an additional manufacturing process of introducing the glue, and there was concern about the consumer's attitude toward a natto product that used raw materials other than soybeans and natto bacteria. There is also the method of reducing natto stickiness by using *okara* rather than soybeans as a raw ingredient,<sup>[3]</sup> but when this method was used, there was no soybean in the raw ingredient and the flavor of natto was lost.

From the above steps, to manufacture natto with low stickiness without additional facility investment, we considered it essential that natto bacteria that could produce natto with low stickiness were bred.

# 4 Breeding method for natto bacteria that could produce natto with low stickiness

The viscous component of natto is mainly composed of a polymer called  $\gamma$ -polyglutamic acid ( $\gamma$ -PGA)<sup>[4]</sup> in which the amino group and the  $\gamma$ -carboxyl group of glutamic acid are condensed, and a polymer called levan<sup>[5]</sup> in which fructose is condensed. Particularly,  $\gamma$ -PGA contributes to the sticky texture, and we considered selecting a strain with low content of this component.

Regarding bacteria or fungi, not limited to natto bacteria, it is known that sometimes the original trait is not maintained and the phenotype may differ from the parent strain when subculturing is repeated. Therefore, to maintain stability in using microorganisms in food, the original strain must be stored in glycerol stocks, liquid-drying tubes, or others, and in general practice, cell expansion is done from the stock close to the original generation. In our research, we conducted an opposite method. Specifically, in repeating the subculturing of the same strain, we isolated all the strains that we felt had altered appearance (that were suspected of natural mutation) compared to the original colony in the culture. Natto was made using all these suspect strains, and the strain from which we were able to create natto with low stickiness was selected (Table 1). For breeding the natto bacteria, culture generally used for natto bacteria was used, including cultures containing LB, E9,<sup>[6]</sup> and soybean powder. In many cases, there was tendency of finding a strain that was different in appearance from the original strain after subculturing about five times. The work process of breeding the natto bacteria with low stickiness is shown in Fig. 2.

As details of the selection method for obtaining the natto bacteria strain with low stickiness are shown in Fig. 2, Miyagino bacteria, which is a commercially-available starter strain, was planted on an agar plate of soybean powder (soybean flour 0.5 g, sodium chloride 0.2 g, glucose 0.1 g, agar 1.5 g, and water 100 ml) and cultured at 37 °C for 48 hours. The grown colony was replanted on a new soybean powder agar plate and cultured at 37 °C for 48 hours. This was repeated five times or more. Replanting was repeated, and the colony grown on a soy powder agar plate was picked, planted on a LB agar plate, and cultured at 37 °C for 24 hours. The colony that appeared different from the original colony was transferred to a new LB agar plate to conduct pure culture. Natto was made from all strains that were isolated. All the candidate strains that were isolated were coated onto Schaeffer agar plates [Nutrient Broth (Difco Laboratories, Ltd.) 1.2 g, magnesium sulfate heptahydrate 0.025 g, potassium chloride 0.1 g, iron (II) sulfate heptahydrate 0.0278 mg, calcium nitrate tetrahydrate 23.6 mg, manganese (II) chloride tetrahydrate 0.198 mg, agar 1.5 g, and water 100 ml], and were cultured at 37 °C for two days to obtain

sporulation. Then, the spores were collected and suspended in sterilized water. After checking the number of bacteria in the adjusted spore suspension, trial manufacture of natto was done.

Manufacturing of natto was done as follows. First, soybeans were soaked in 20 °C water three times the amount of soybeans for 16 hours, drained, and cooked for 30 min at 0.18 MPa. Next, natto bacteria spore suspension was sprayed, so the number of aforementioned soybean bacteria will be 10<sup>3</sup> spores per 1 g cooked soybeans. This was stirred well. A set amount of the mixture was placed in a foam-polystyrene container, and was covered with a polyethylene film with small holes. Next, a fermentation process was done at 39 °C and humidity of 90 % for 18 hours, and then, cooling was done at 20 °C and humidity of 50 % for 2 hours. Curing was done at cool storage of 5 °C for 1 day or more, to obtain natto.

Normally, fermentation is done for about 18 hours, but less sticky natto could be manufactured even when longer fermentation hours were taken. For the natto thus manufactured, coverage of bacteria, hardness of beans, and stickiness were evaluated, and natto bacteria strains with less stickiness compared to conventional natto strains were selected. The selected bacteria strains were subjected to 16S rRNA genetic analysis and a biotin auxotrophy test, and from those results, it was confirmed that the natto bacteria belonged to the *Bacillus subtilis* group.

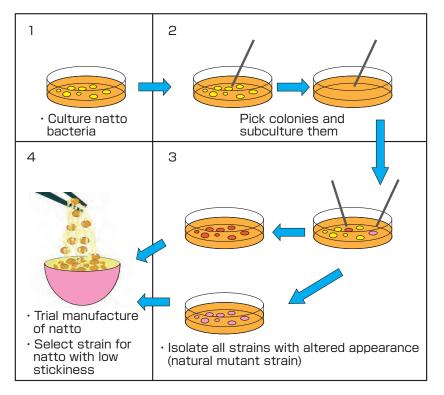


Fig. 2 Breeding method of natto bacteria to manufacture natto with low stickiness

The difference between natto bacteria and non-nattoproducing *Bacillus subtilis* is that natto bacteria show biotin auxotrophy and have the ability to produce viscous substances, while non-natto-producing *Bacillus subtilis* do not show such characteristics.

Since we screened all strains that seemed to have changed appearance, we had about a total of 100–200 strains.

Natto was created from all such strains, and the strains with low stickiness compared to the original strain were confirmed for several strains, but there were variations. Among those, one strain that had particularly low stickiness, excellent mouth-feel, and taste was selected (Fig. 3). This strain was named IBARAKI *lst-1* and was used for further manufacturing and testing.

# 5 Evaluation method for the characteristics of natto with low stickiness manufactured using the IBARAKI *l*st-1 strain

# 5.1 Measurement of viscous substance production in natto

Natto was manufactured using the IBARAKI *l*st-1 and Miyagino strains to produce natto that would serve as samples. The natto (about 13 g) was weighed and placed in 50 ml centrifugal tubes, and 5 ml of 10 mmol/L sodium phosphate buffer (pH 7.0) containing 1 mol/L sodium chloride was added.

After mixing and cleansing the soybean surface, the samples were centrifuged for 5 min at 8,700×g and 4 °C, and the supernatant was batched off into new 50 ml centrifugal tubes. This was repeated three times, and a water-soluble substance was collected. The collected water-soluble substance was centrifuged for 20 min at 8,700×g and 4 °C, and the process of collecting the supernatant was repeated twice. To the 10 ml supernatant obtained, 25 ml of 99.5 % ethanol was added. This was centrifuged for 10 min at 8,700×g and 4 °C, to collect a viscous substance. The collected viscous substance was dried under reduced pressure, and was weighed. The test was done in three series, average values and standard deviations were calculated, and the values were compared.



Fig. 3 Natto bacteria with low stickiness (left) and natto made from existing natto bacteria strain (right)

# 5.2 Maximum viscosity measurement when natto was stirred

The Rapid Visco Analyzer RVA-4 (Newport Scientific, Inc.) was used to investigate natto viscosity, and mixing resistance was measured. Since the mixing resistance of natto itself could not be measured since the viscosity was too high, the measurement sample was prepared by adding 1 ml of water to 10 g of natto. To measure the mixing resistance, the paddle and cup exclusive of RVA-4 were used. The measurement of maximum viscosity was done at 180 rotations per min, sample temperature of 20 °C, and measurement time of 2 min. The measurements were taken in four series, average values and standard deviations were calculated, and the values were compared.

### 5.3 Measurement of natto hardness

The hardness of natto was measured as described in a previously published paper.<sup>[7]</sup> For about 50 beans of natto, the breaking strength (hardness) in the short-axis direction was measured. Ten beans each with maximum and minimum values were eliminated from the data, and the average values and standard deviations were calculated for the intermediate 30 beans, and this was used as measurement results.

# 5.4 Color measurement of natto

The color of natto was measured using the spectrophotometric color-difference meter SE-2000 (Nippon Denshoku Industries Co., Ltd.). The average values of  $L^*a^*b^*$  measurements of 10 beans were calculated and used as the measurement value. L\* represents brightness (value range of 0 to 100), and the larger value indicates that it is a bright color. Values for a\* (value range of -60 to +60) and b\* (value range of -60 to +60) represent chromaticity, and higher values of a\* show that red is strong, while higher values of b\* show that yellow is strong (Fig. 4).

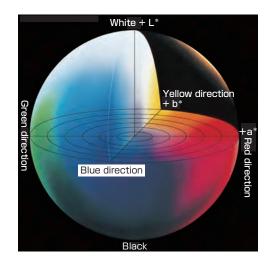


Fig. 4 Relationship between brightness L\* and chromaticity a\* b\* From *Iro Iro Zatsugaku*, Konica Minolta Japan Inc.

# 5.5 Measurement of contents of basic nutrients, vitamin K, and polyamine in natto

The basic nutrients, vitamin K that is found abundantly in natto, and polyamines<sup>[8][9]</sup> (putrecine, spermidine, and spermine) that are drawing attention as natto nutrients were analyzed. The analysis of basic nutrients and vitamin K were conducted according to previously published papers,<sup>[10][11]</sup> and the analysis of polyamine was done as follows. Natto was manufactured using the same method as described in Chapter 4, and it was freeze-dried and crushed into powder with a mill. Then 5 ml of 5 % trichloroacetic acid was added to a 1 g sample, and this was mixed in the vortex mixer. This was centrifuged at 8,700×g and 4 °C for 10 min, and the supernatant was collected. After repeating this process three times, it was diluted in a graduated flask to a 25 ml solution. To 50  $\mu$ l of diluted extract, 50  $\mu$ l of 6 nmol/ml 1,7-diaminoheptane/0.1 M hydrochloric acid solution, 200  $\mu$ l of sodium carbonate saturated solution, and 200  $\mu$ l of 10 mg/ml dansyl chloride/acetone solution were added. Using a heat block, this was cultured at 70 °C for 15 min. After adding 25  $\mu$ l of 100 mg/ml L-proline, this was cultured at 70 °C for 5 min. After adding 500  $\mu$ l of toluene and mixing well, 500  $\mu$ l of supernatant was extracted. After drying under reduced pressure, this was dissolved in 800  $\mu$ l of acetonitrile, filtered with a 0.45  $\mu$ m membrane filter, and used as the analysis sample. The polyamine analysis was done by highperformance liquid chromatography (HPLC). As a mobile phase, 10 mM ammonium dihydrogenphosphate (liquid A) and acetonitrile (liquid B) were used. Starting from a state in which the ratio of liquid A to B was 45:55, the analysis was conducted under a condition where the ratio of liquid B would increase linearly during the analysis time. The flow rate was set at a constant 0.9 ml/min.

The ODS column was used, and the column oven was set at 50 °C. A fluorescence detector was used for detection, and the excitation wavelength was set at 340 nm and fluorescent wavelength was 515 nm. The polyamine content in the sample was calculated from the calibration curve using the reference standard.

#### 5.6 Sensory test of natto

The comparative evaluation of the two types of natto was conducted by 38 healthy males and females, in the 10s to 60s age group, without providing any information about the natto. The natto manufactured by the Miyagino strain was set at 3 points (normal) for all evaluation items, and the characteristics and properties of natto manufactured by the IBARAKI *lst-1* strain were compared and evaluated. Six evaluation items were set, and questions were asked in a multiple choice form for each item of whether natto made from the Miyagino stain was better (stronger), no difference, or natto made from the IBARAKI *lst-1* was better (stronger).

### Table 2. Comparison of viscous substance production (unit: mg/g natto)

IBARAKI lst-1	Miyagino
11.5±0.6	15.3±0.4

Table 3. Comparison of maximum viscosity when natto is stirred (unit: RVU)

IBARAKI &st-1	Miyagino
201±49	582±27

#### Table 4. Comparison of natto hardness (unit: g)

IBARAKI &st-1	Miyagino
85.2±8.7	88.9±9.5

# 6 Evaluation of characteristics of natto with low stickiness and discussion

# 6.1 Comparison of production of viscous substance in natto

The amounts of viscous substance produced were compared for natto manufactured from the IBARAKI *lst-1* strain and from the Miyagino strain which is a commercially-available starter strain. As a result, it was shown that with the natto of the IBARAKI *lst-1* strain, production of viscous substances decreased about 25 % compared to that of the Miyagino strain (Table 2).

From this, it was confirmed that IBARAKI *lst-1* was capable of making natto with less stickiness compared to the Miyagino strain.

# 6.2 Comparison of maximum viscosity when natto is stirred

It was found that the maximum viscosity when the natto made from IBARAKI *lst-1* was stirred decreased 65 % or more compared to the natto manufactured by the Miyagino strain.

From this, it was indicated that the natto made from IBARAKI *lst-1* flowed well, and was easier to handle and swallow compared to the natto made from the Miyagino strain (Table 3).

#### 6.3 Comparison of natto hardness

The average values of the hardness of natto manufactured by Miyagino and IBARAKI (st-1 strains (Table 4) were processed statistically, t test was done at P value 0.05, and the result was 0.119. Since this value was greater than 0.05 (P value), there was no significant difference in the hardness between the two kinds of natto. Therefore, it was confirmed

#### Table 5. Comparison of natto color

Color/natto	IBARAKI &st-1	Miyagino
L*	55.14	58.97
a*	5.49	4.55
b*	11.90	10.32

### Table 6. Comparison of basic nutrients of natto (per 100 g)

	Energy (Kcal)	Moisture (g)	Protein (g)	Fat (g)	Carbohydrate (g)	Ash (g)
IBARAKI &st-1	201	58.6	16.2	9.7	13.2	2.3
Miyagino	197	59.2	16.3	9.5	12.7	2.3

# Table 7. Comparison of vitamin K content in natto (unit: $\mu$ g/100 g natto)

Bacteria species/	Vitamin $K_1$	Vitamin K <sub>2</sub>	
vitamin K type	Phylloquinone	Menaquinone-4	Menaquinone-7
IBARAKI &st-1	22.1±0.8	3.6±0.4	1101±38
Miyagino	22.5±0.2	1.5±0.0	1027±14

from the measurement data that almost the same hardness would be obtained for the natto manufactured using the two strains.

#### 6.4 Comparison of natto color

It was shown from the data that the two kinds of natto had almost the same color (Table 5).

# 6.5 Comparison of contents of basic nutrients, vitamin K, and polyamine in natto

The analysis results of the basic nutrients, vitamin K, and polyamine in natto are shown in Tables 6–8. It was shown that there were hardly any differences in the basic nutrients and vitamin K in the nattos manufactured from IBARAKI  $\ell$ st-1 and Miyagino strains, and there was no nutritional difference between the two. When the natto bacteria is cultured in synthetic culture containing no polyamine at all and the polyamine content in the culture liquid is analyzed, spermine is not detected.

The natto bacteria mainly biosynthesizes spermidine. Looking at the result of Table 8, the spermidine content in the Miyagino strain natto was  $30.6 \pm 0.7 \ \mu$ g per 1 g dried natto, while it was  $30.6 \pm 0.2 \ \mu$ g in IBARAKI (st-1 strain natto. As shown in Tables 6–8, in terms of nutritional content of natto, it was shown that the IBARAKI (st-1 strain was not inferior to the Miyagino one, which is the commercially-available starter strain.

Table 8. Comparison of polyamine content in natto (unit:  $\mu$ g/g dried natto)

Polyamine type/ bacteria species	IBARAKI १st-१	Miyagino
Putrescine	2.4±0.1	2.4±0.1
Spermidine	30.6±0.2	30.6±0.7
Spermine	7.9±0.1	7.3±0.2

### 6.6 Results of the sensory test for natto

The evaluation results of natto by a 5-point method and the evaluation results of natto by a multiple choice method are shown in Table 9 and Table 10, respectively. Looking at Table 9, the evaluation of the stickiness item was 3 points for the Miyagino strain natto and 1.47 points for the IBARAKI *l*st-1 one, and it became clear that the difference between the two was recognized.

In the items other than stickiness, excluding the "coverage by bacteria" that indicates the growth of the bacteria, there was no weakness against the Miyagino strain natto and some items had better evaluation. Also, looking at Table 10, pertaining to stickiness, 38 out of 38 people all answered that the Miyagino strain natto was stronger. This means that 100 % of the people felt that the IBARAKI {st-1 strain natto had less stickiness, and it was shown that there was a clear difference against the Miyagino one.

For the question, "which is easier to swallow," six people answered that the natto made from the Miyagino strain was easier to swallow, while 24 people or four times that number chose the IBARAKI  $\ell$ st-1 strain natto.

For the question, "which was better for care food considering ease of swallowing and handling," 32 out of 38 people answered that the IBARAKI  $\ell$ st-1 strain natto was more suitable. From the evaluation results of Table 9 and 10, the

			Natto manufactured by IBARAKI &st-1	Natto manufactured by Miyagino
1. Coverage of bacteria	ly bad Normal Slightly good	Good	2.70±0.67	3.00
2. Bacteriolytic condition	ly high Normal Slightly low	Low 5	3.47±0.74	3.00
and crushing	ly high Normal Slightly low	Low 	3.47±0.76	3.00
4. Color of beans	ly bad Normal Slightly good	Good 5	3.23±0.75	3.00
5. Smell	ly bad Normal Slightly good	Good 5	3.23±0.93	3.00
6. Hardness	ly hard Normal Slightly soft	Soft	3.27±0.97	3.00
7. Taste	ly bad Normal Slightly good	Good	3.13±0.92	3.00
8. Stickiness	y weak Normal Slightly strong	Strong	1.47±0.55	3.00
9. General	ly bad Normal Slightly good	Good 5	3.05±0.87	3.00

Table 9. Results of sensory test for natto 1

#### Table 10. Results of sensory test for natto 2

	Natto manufactured by IBARAKI &st-1 (no. people)	Natto manufactured by Miyagino bacteria (no. people)	No difference between the two (no. people)
Which has better taste?	13	14	11
Which has better smell?	11	9	18
Which has stronger stickiness?	0	38	0
Which is easier to swallow?	24	6	8
In general, which is better?	14	16	8
Which is better as care food?	32	5	1

natto manufactured from the IBARAKI lst-1 strain was not inferior to the conventional natto in quality including taste, and differed only in the point that it had low stickiness.

To stably obtain natto with low stickiness even when IBARAKI lst-1 strain was used repeatedly in natto manufacturing, the original strain was stored by freezing, expansion was done from that strain, and care was taken so that change in quality would not occur through subculturing.

To maintain the low stickiness of natto at the company production sites, it is necessary to pay attention so that there will be no contamination by other natto bacteria within the manufacturing plant.

# 7 Development of the research result

#### 7.1 Collaboration of industry-academia-government

Ibaraki ITI called out to the natto companies in Ibaraki that wished to sell the natto made by the IBARAKI *lst-1* strain to foreign countries, through the Ibaraki Council for the Promotion of Growing Industry.

Specifically, the coordinator of the Tsukuba Center Inc. approached the companies that were interested in product realization of natto with low stickiness, and Ibaraki ITI distributed IBARAKI *l*st-1 to the companies with which agreements were made and provided advice on manufacturing, in collaboration with the Tsukuba Center.

The Industrial Policy Division of Ibaraki prefectural government asked Associate Professor Tadanobu Hara, who was studying visual design and package design at the Faculty of Art and Design, the University of Tsukuba, to design the logo. The Division also asked Mr. Hiroshi Fujiwara, a food analyst working as food advisor at the Agriculture, Forestry and Fisheries Project of Ibaraki prefectural government, for advice on the brand strategy. It also asked Mr. Yoshinaga Jinbo, who was the chef and owner of Hatake Aoyama, a restaurant in Tokyo, to develop natto cuisine recipe. Mr. Jinbo was from Hitachi, Ibaraki Prefecture and had studied cooking in France and Italy.

The Industrial Policy Division and Ibaraki ITI built the following industry-academia-government collaboration system, while meeting occasionally to decide on participation in overseas exhibitions and management of intellectual property of natto bacteria that could manufacture lowstickiness natto, as well as on how to carry on the project if there were differences in opinions among the natto companies in Ibaraki (Fig. 5).

Within this industry-academia-government collaboration system, Associate Professor Hara of the University of Tsukuba proposed a unified brand name and logo, "Mamenoka" (Fig. 6). The definition of "Mamenoka" was set as "it must be natto that used IBARAKI {st-1."

For use in processed food, Chef Jinbo created menus using Mamenoka (Fig. 7).

# 7.2 Participation in food exhibitions in Japan and overseas

The Ibaraki Council for the Promotion of Growing Industry established the "Mamenoka Project" that is a project for selling the natto manufactured using IBARAKI *lst-1* as a new food ingredient, and participated in the following food exhibitions.

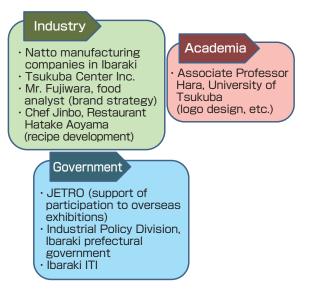


Fig. 5 Collaborative relationship of industry-academiagovernment

- Sirha 2015 (Salon International de la Restauration, de l' Hotellerie et de l'Alimentation)
- Held in Lyon, France on January 24–28, 2015 (Fig. 8). (2) Foodex Japan 2015

Held at Makuhari Messe on March 3–6, 2015 (Fig. 9). (3) Anuga FoodTec 2015

- Held in Koeln, Germany on October 10–14, 2015 (Fig. 10). (4) Foodex Japan 2016
  - Held at Makuhari Messe on March 8-11, 2016.

After the exhibitions, some business negotiations are in progress.

While we attended domestic food exhibitions, due to circumstances, we did not go to overseas exhibitions. However, from the stories of the people who participated in the overseas exhibition, the Mamenoka samples were taken very favorably both in France and Germany, and many people of importing companies, restaurant industries, and food manufacturers sampled the product.

However, since foreigners do not have the custom of eating natto with rice like the Japanese, nor do they have the cold chain for natto in food distribution as in Japan, to pioneer



Fig. 6 Unified logo for MAMENOKA



**Fig. 7 Example of cuisine using MAMENOKA** Boule de Natto (natto is added to fermented butter, and échalot, wasabi, and konbu powder are used as secret ingredients)

the overseas sales route for natto, "it is necessary to create new natto products such as dip sauce, paste, or dressing, to propose new ways to eat it, and to develop technologies that allow the natto product to be exposed to room temperature and to lengthen the open date."

Currently, Ibaraki ITI continues to provide technical support to the natto companies in Ibaraki Prefecture. Also, it is working on breeding research for new natto bacteria that allows the extension of the open date of natto products.

# 7.3 Intellectual property rights for natto bacteria that can make low stickiness natto

The natto bacteria that can make natto with low stickiness



Fig. 8 People visiting the MAMENOKA booth at Sirha 2015



Fig. 9 Exhibition booth at Foodex Japan 2015



Fig. 10 Sampling of MAMENOKA products at Anuga 2015

was named IBARAKI *l*st-1, and the patent was obtained as "Name of invention: Natto bacteria strain with low stickiness, manufacturing method for natto using this natto bacteria strain, and the natto" (Patent No. 5754009, filed June 5, 2015).

This natto mutant strain was sent to the Patent Microorganism Depository of the National Institute of Technology an Evaluation, under Accession Number NITE P-01836.

IBARAKI lst-1 has been provided to six companies in the Ibaraki Prefecture that are participating in the Mamenoka Project, as of August 1, 2017.

### 7.4 Mass media coverage of the results

To publicize the prefectural support of overseas diffusion of natto, the Industrial Policy Division held a natto cuisine tasting event for foreigners living in Ibaraki. Mass media were approached to cover this tasting event, and with the coverage by the Mito Broadcasting Station of Japan Broadcasting Corporation (NHK) that was seeking local news, the Mamenoka project was reported in the news programs of various commercial stations such as TV Tokyo, as well as by NHK Main Station. The Mamenoka project received the Broadcasting Culture Award for the Kanto-Koshinetsu region from NHK. This is an award for persons (organizations) that contributed to the development of local culture, living, or industry throughout the year in the Kanto-Koshinetsu region, or the persons (organizations) that contributed to various activities of NHK, and it was the first time in four years for an entity of Ibaraki to receive this award. Thus, the Mamenoka project received wide attention and was acclaimed highly in society.

Ibaraki ITI told the mass media reporters, "Please cover the companies manufacturing Mamenoka in the Ibaraki Prefecture, not just ITI," and handed them a "list of companies manufacturing Mamenoka" to help expand the sales route of the Mamenoka product.

Later, some of the Mamenoka manufacturers were covered by the mass media, but the companies were not covered equally, and most of the coverage focused on a certain company only. When the prefectural government and the private companies start up a project in collaboration, the future issue is to obtain understanding and cooperation of the mass media to ensure that they do not focus on a single company only.

# 7.5 Product development by companies participating in the Mamenoka project

As of August 1, 2017, Mamenoka is produced by five companies, Asaichiban Corporation, Kanasago Corporation, Y. K. Kikusui Shokuhin, Y. K. Toko Foods, and Higeta Shokuhin Co., Ltd. The products can be purchased through their websites or at Ibaraki Marché (a shop in Tokyo that sells products of Ibaraki Prefecture). The companies differentiated their products from each other through selection of raw material soybeans, manufacture methods, and packaging (Fig. 11).

Due to the low stickiness, Mamenoka has the merit that it can be easily processed such as grinding into paste. Utilizing this merit, the participating members of the Mamenoka project worked on processed products using Mamenoka, and developed dip sauce that could be used for vegetables, waffles in which Mamenoka was kneaded into the dough, Mamenoka paste that could be spread on bread, Mameoka dressing, and others (Fig. 12).

The project members are working on new processed products, and Ibaraki ITI is supporting the development through quality evaluation including bacteria testing.

New processed products are being developed through collaboration with other types of businesses. There is development of *gyoza* (dumplings) containing Mamenoka at ramen noodle stores, as well as chocolate cakes, madeleines, scones, and others using Mamenoka by confectionery companies. In October 2015, there was a trial of providing dry curry containing Mamenoka as school lunch at Bando City, Ibaraki Prefecture. The use of Mamenoka is spreading to various places.

Although the Mamenoka Project was effective in pioneering the domestic market for natto products by the Ibaraki companies, further administrative support is necessary for the effect to take place in overseas markets. Currently, the



**Fig. 11 MAMENOKA from companies participating in the MAMENOKA project** From the website pages of MAMENOKA companies

Industrial Policy Division of Ibaraki prefectural government is hiring people of private companies with rich experience in trading overseas. They are stationed in Paris and are approaching the local buyers for products from Ibaraki as well as natto.



Fig. 12 Examples of processing using MAMENOKA From the homepages of MAMENOKA companies

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# Authors

# Yuji Kubo

Chief, Local Food Division, Industrial Technology Institute of Ibaraki Prefecture (Ibaraki ITI). Graduated from the Faculty of Agriculture, Tohoku University in 2004. Completed the master's program at the Graduate School of Life Sciences, Tohoku University in 2006. Employed as staff (chemistry) of Ibaraki Prefecture in 2006, and was assigned to Ibaraki



ITI. Was in charge of food testing research mainly for natto. Obtained doctor's degree (agriculture; doctoral degree by thesis) at the United Graduate School of Agricultural Science, Tokyo University of Agriculture and Technology in 2014, upon summarizing the research results on natto that was conducted at Ibaraki ITI. Writings include "Chapter 3: Daizu kako shokuhin no kinosei (Functionality of processed soybean product)" in *Daizu No Eiyo To Kinosei* (Nutrition and Functionality of Soybeans), CMC Publishing, 2014. Received the Broadcasting Culture Award for Kanto-Koshinetsu region, Japan Broadcasting Corporation (NHK), 2016.

#### **Rikio** NAKAGAWA

Director, Local Food Division, Industrial Technology Institute of Ibaraki Prefecture (Ibaraki ITI). Graduated from the Department of Agricultural Chemistry, Faculty of Agriculture, The University of Tokyo in 1986. Employed as staff (chemistry) of Ibaraki Prefecture in 1986, and was assigned as the Food Sanitation Inspector at the Mitsukaido



Public Health Center (currently Joso Public Health Center), Ibaraki Prefecture. Was in charge of acreage reduction policy for rice and subsidiaries for soil improvement, at the Agricultural Department of Ibaraki prefectural government from 1988; was in charge of manufacturing dried plums from frozen plums and CA storage test of fruits, etc. at the Distribution Processing Laboratory, Horticultural Research Institute, Ibaraki Agricultural Center from 1993; was in charge of preventing discoloration of lotus roots, comparison of processability into tofu of different soybean species, senbei manufacturing test using low-amylose rice, etc. at the Processed Food Division (current Local Food Division), Ibaraki ITI; Chief Researcher in 2003; Director, Local Food Division in 2007; and also Principal Researcher since 2014.

# **Discussions with Reviewers**

#### 1 Overall

#### Comment (Keiichi Ikegami, AIST)

This paper discusses the case in which attempt was made for overseas diffusion of natto, a local product, by integration of biotechnology, marketing, and cooking techniques, through wide-ranging collaboration of industry-academia-government personnel. The fact that the products have been actually realized by multiple companies supports the importance for the researchers of public research organizations and the people of industry and academia to share the "scenario" for innovation. I think this is a case study that should be discussed in *Synthesiology*.

# Comment (Noboru Yumoto, National Cerebral and Cardiovascular Center)

This paper describes the process by which natto with low stickiness was developed for overseas diffusion and was made into a brand through the collaboration of industry-academia-government. Not being bound by the Japanese custom of natto consumption, there was a clear scenario, and many companies were able to achieve product realization. I think the paper is suitable for publication in *Synthesiology*.

### 2 Clarification of the scenario

#### Comment (Noboru Yumoto)

For *Synthesiology*, the requirement of a research paper is the authors' originality of the scenario (the process by which the elemental technologies necessary to achieve the goal were selected and integrated). Although the current manuscript enables the reader to grasp the overall picture, it is slightly difficult to see how the elements were synthesized according to the authors' scenario. Can you create a flowchart of the scenario and clarify how the elemental technologies were synthesized? Also, can you include a description that emphasizes the scenario in your

### abstract?

## Answer (Rikio Nakagawa)

I created a flowchart of the scenario as Fig. 1 to clarify how the elemental technologies were synthesized. For the abstract, I summarized and added the part about "breeding of the natto bacteria" and "evaluation of the characteristics of natto manufactured with the bred natto bacteria," and also changed the description to emphasize the scenario for overseas diffusion.

### 3 Building of collaborative relationship

### Comment (Keiichi Ikegami)

You mention the building of collaborative relationships among wide-ranging organizations and people. I think such building of organic collaborative relationships is extremely interesting to the readers of Synthesiology. Please add how such collaborative relationships were built and how your "scenario" was shared. Answer (Rikio Nakagawa)

For the building of collaborative relationships, I added to the text to the extent that can be disclosed.

#### 4 Subculturing and evaluation of characteristics Question (Noboru Yumoto)

The natto bacteria you obtained in this research was obtained through repeated subculturing. How do you maintain stability of the bacteria during a repeated use? Also, what was the degree of deviation of the results for the sensory test?

#### Answer (Rikio Nakagawa)

For the repeated use of natto bacteria with low stickiness, we ensured that changes in quality did not occur during subculturing by freeze-storing the original strain and expanding from the original. For stability, I added a simple explanation to the paper. For variations in the results of the sensory test, I added the value of sample standard deviation ( $\sigma_{n-1}$ ) in Table 9.

#### Comment (Keiichi Ikegami)

Are the people who participated in the sensory test people of the natto industry? Do they like eating natto in the first place? Compared to the general sensory test, I think this is a case where individual preference has major effect on the results, so I would like to know who the testers were.

### Answer (Rikio Nakagawa)

In the stage of natto bacteria breeding research, we had people

of the natto companies in Ibaraki do the sensory test. However, the sensory test in Subchapter 5.6 was done by a panel of the following people.

[Sensory test panel]

• Staff of Food Research Institute, National Agriculture and Food Research Organization

- · People of natto companies in Ibaraki Prefecture
- Staff of Industrial Policy Division, Ibaraki prefectural government
- Friends and acquaintances of the authors

We did not question whether the panel members liked or disliked natto. However, the members live in Ibaraki, so I think there was "a mixture of people who like natto and people who are neutral about natto."

#### **5 Future efforts**

#### Comment (Keiichi Ikegami)

You write about participation in several fairs and exhibitions, but there is diversity in what you call "overseas," and I imagine that there are differences according to countries or ethnicities. What were your impressions from participating in the exhibitions, and how did you feedback the impressions to product development? I think these are points in which the Synthesiology readers will be interested, so please make some additions. Comment (Noboru Yumoto)

In the final part, you write, "the use of Mamenoka is spreading to different kinds of food." If you have any activities that you have in mind that are different and unique from what you have been doing, can you explain that to the extent that you can? Particularly, can you describe activities that are necessary to further promote branding and international diffusion that emerged through various discussions at the exhibitions and in making various products?

#### Answer (Rikio Nakagawa)

Some of these are business matters and I cannot talk about the details, but I added to Subchapter 7.2 my impressions from the exhibitions, as well as the direction of technical support such as "breeding research of natto bacteria that may extend the open date" that is being conducted by budget from a different project, and to Subchapter 7.5 the efforts of companies to which the technology was provided and our administrative support.

# Submarine earthquake- and tsunami-induced event deposits

# Disturbance of the sea floor by huge earthquakes and their related tsunamis, and the use of disturbance records in marine sediments for the history of past huge earthquakes and tsunamis—

# Ken IKEHARA<sup>\*</sup> and Kazuko USAMI

#### [Translation from Synthesiology, Vol.11, No.1, p.12-22 (2018)]

Huge earthquakes and tsunamis have agitated and disturbed the sea floor. Many marine geological surveys after the 2011 off the Pacific coast of Tohoku Earthquake indicated large disturbances of the sea floor by the earthquake and its related tsunami across a wide area from the coastal to the Japan Trench floor. Resuspension of marine surface sediment by the earthquake and tsunami might generate turbidity currents. Deposition of turbidites, which are deposits from turbidity currents, has been recognized. Therefore, earthquake- and tsunami-induced turbidity of past huge earthquakes and tsunamis. For the estimation of the origin and evolution of earthquake- and tsunami-induced turbidity currents and the selection of suitable locations for turbidite paleoseismology, marine geological information such as samples and characteristics of surface sediments and depositional modes is useful and important.

Keywords: Marine sediment, sea floor environments, turbulence, earthquake, turbidite, marine geological map

# **1** Introduction

The 2011 off the Pacific coast of Tohoku Earthquake (hereinafter, will be called 2011 off Tohoku Earthquake) that occurred on March 11, 2011 and the giant tsunamis that followed caused serious damage to the coastal areas from East Japan to Hokkaido, with the worst damage on the Pacific coast of the Tohoku region. This earthquake and tsunamis were initially labeled "beyond expectation," but it is now known that the tsunami deposits on land show that there were previous cases observed in stratigraphic records,<sup>[1]</sup> and it was recognized that analysis of records is important in understanding the history of giant earthquakes and tsunamis. On the other hand, it was revealed that the results of analyzed records had not been utilized sufficiently in disaster prevention plans.<sup>[2]</sup> Some of the tsunami deposits on land may contain particles originating from deep water regions from which they cannot be transported by ordinary or stormy waves,<sup>[3]</sup> and it is thought that there are incidences where marine sediment is disturbed by tsunamis and washed ashore. In fact, according to the results of surveys of the Tohoku coast and offshore regions after 2011 that will be mentioned later, the phenomena of resuspension, reworking, and redeposition of the marine sediments have been reported in wide-ranging areas from the Sanriku coast forearc slopes to the Japan Trench floor, including the coasts and the shelf regions from the Sendai Bay, Sanriku, and off Hachinohe.<sup>[4]-[9]</sup> This indicates that in places where such redeposition phenomena remain, the evidences of past giant earthquakes and tsunamis may remain in the geological record on the sea floor, just like the tsunami deposits on land, and there is a possibility

that these can be used for decoding history. In fact, the past earthquake occurrence history is being studied by comparing the tsunami deposit records on land with the sediment records in the Japan Trench floor.<sup>[7]</sup>

On the other hand, the areas from the shore to the offshore are closely intertwined with human activities, such as being used for constructions, fishing, and leisure. Moreover, fishing activities are conducted in the deepwater area from the shelf to the upper slopes further off the coast, and to know how the submarine environment from the shore to the offshore changes by earthquakes and tsunamis is important in predicting how earthquakes and tsunamis may affect human activities. However, there are very few cases that specifically show how the submarine environment changed due to certain earthquake and tsunami events or how the environment recovered.

Here, based on the current status of research after the 2011 off Tohoku Earthquake and Tsunami, we shall summarize the disturbance of the sea floor due to earthquakes and tsunamis and the process of resuspension and redeposition of marine sediment due to such events. After outlining the current situation of earthquake recurrence history research using the deposits produced during the earthquake (hereinafter, will be called earthquake event deposits), we wish to emphasize the importance of organizing marine sediment information as fundamental information that allows accurate understanding of the process. Also, we discuss what should be done for the future of paleoseimological research using earthquake and tsunami records left in marine sediment.

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# 2 Reworking, resuspension, and redeposition of marine sediment by earthquakes and tsunamis and the changes in marine environment

The occurrence of large-scale transport of sediments on the sea floor due to an earthquake became apparent during the Grand Banks Earthquake (M7.2) that occurred on the Atlantic coast of North America in 1929. Concerning this earthquake, several ocean cables that were installed in the submarine slope were severed, and the occurrence of turbidity currents was assumed due to the collapse of submarine slopes.<sup>[10]</sup> A turbidity current is a gravity current that flows down a slope due to the force of gravity while the particles are maintained in the turbulence of seawater mixed with sediment particles<sup>[11]</sup> (Fig. 1A). The speed of the turbidity currents of the Grand Banks Earthquake was the fastest in the slope region at 28.4 m/s, calculated from the distance between the ocean cables and the disconnect time, and was estimated to be about 8.3-6.2 m/s at the deep sea basin floor further off the coast.<sup>[10]</sup> Later, turbidites (deposits of the turbidity currents, Fig. 1B) were found in areas where the turbidity currents flowed, and it was found that a series of events occurred: collapse of submarine slopes due to the earthquake  $\rightarrow$  occurrence and flow of turbidity currents  $\rightarrow$  deposition of turbidites.<sup>[12]</sup> Similar sequential severance of ocean cables during earthquakes have been reported in several cases,<sup>[13][14]</sup> and this also occurred during the 2011 off

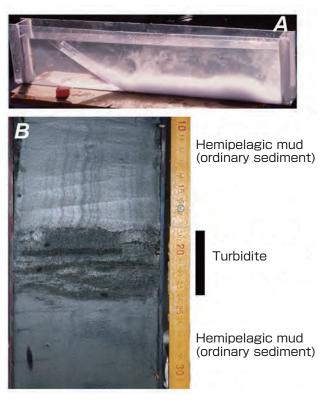


Fig. 1 Turbidity current (A) created in a water tank, and turbidite (B) that is a deposit from turbidity current as seen in a marine sediment core

Tohoku Earthquake.<sup>[15]</sup> There are many reports of turbidite depositions on sea floors around epicenters in submarine surveys immediately after earthquakes,<sup>[16]–[18]</sup> and it is clear that earthquakes are one of the generative factors of turbidites.

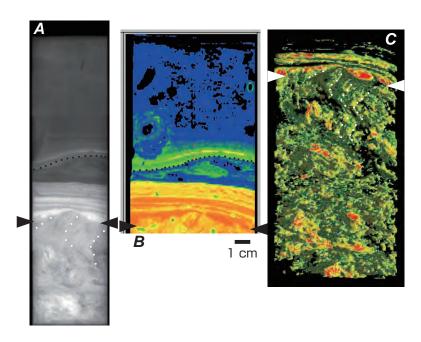
It has become clear from several reports that the 2011 off Tohoku Earthquake and Tsunami had major effect on the sea floor. First, the changes in the coastline due to crustal movements following the earthquake and tsunamis could be seen easily by the naked eye, aerial photos, and satellite images. Many reports were provided by the mass media and others immediately after the earthquake. The topographical change of the coastal region adjoining the seashore was estimated from the bathymetric survey in the Sendai Bay, and the geographical distribution of the deposition and erosion of deposits, along with the data for seashore areas, were estimated.<sup>[19]</sup> The sampling of marine sediments showed that the deposits thought to originate from the 2011 off Tohoku Earthquake and Tsunami were thick in places estimated to be deposition areas and thin in places estimated to be erosion areas, and it was found that the results of the bathymetric survey and sediment sampling basically matched well.<sup>[20]</sup> In the southern part of the Sendai Bay, the presence of tsunami deposits on the sea floor was reported through high-resolution seismic surveys and sediment cores.<sup>[21]</sup> On the other hand, in the inland bay that comprises the Sanriku ria coast, the formation of landform due to tsunami deposits is reported at the sea floor of the mouth of the Kesennuma Bay.<sup>[22]</sup> It is also known that the formation of tsunami deposits occurred in the Hirota Bay, the Okirai Bay, the Touni Bay, and others.<sup>[23]</sup> In places such as the Onagawa Bay that is comprised of muddy sediments, tsunami deposits exist as clear sand layers, and their disruption by the activities of benthic organisms after deposition have been observed.<sup>[24]</sup> Sand layers that are intercalated in similar mud layers had been reported before the 2011 earthquake in the northern part of the Sendai Bay, and had been thought to be storm sand layers.<sup>[25]</sup> However, considering the survey results after 2011, it is highly possible that part of the sand layers might originate from tsunamis. In the shelf beyond the Sendai Bay, a deposit layer formed by repeated turbidity currents has been reported.<sup>[6]</sup> The series of fine-grained turbidites observed here are composed of a two-story structure of finegrained turbidites that do not contain cesium 134 in the lower unit, but fine-grained turbidites containing cesium 134 in the upper unit (Fig. 2A, B). It is thought that the lower unit was deposited immediately after the earthquake and tsunamis, while the upper unit was deposited at least a few days after the earthquake and tsunamis, after the Fukushima Daiichi Nuclear Power Plant accident. These indicate that the interior of the Sendai Bay was under a condition in which sediment was suspended for several days or several dozens of days after the earthquake and tsunamis, although details remain unclear, and turbidity currents occurred during the aftershocks following the main quake. The phenomena of

transport and redeposition of sediment in the shallow sea were not limited to the Sendai Bay or off Sanriku that are close to the epicenter. Toyofuku et al.[8] reported that the reworking of sediments occurred down to about a depth of 200 m off Hachinohe. Since the tsunami affects a wide area, it can be assumed that the affected area of the sea floor is also wide. In off Sanriku, it was shown that the shelf sediments were transported to the deep sea area of a depth of about 900 m, from the analysis of benthic foraminifera.<sup>[26]</sup> Interestingly, the turbidites here also had a two-story structure, and the lower turbidites originated from almost the same depth zone in the proximal area while only the topmost upper turbidites contained the shallow water benthic foraminifera. This was interpreted as meaning that the lower turbidites were formed through earthquake disturbance while the upper turbidites were formed by the disturbance in the shallow sea region by the following tsunamis. That is, although the deposit layer seems contiguous, they may have been formed from several different depositional processes by the earthquake and the following tsunamis. The turbidity currents washed away the observation devices set on the sea floor, and caused trouble by clogging the devices with mud.<sup>[5][27]</sup> As it can be seen, the characteristic of a giant tsunami is that it causes wideranging disturbance in the shallow sea offshore of the land where the tsunami strikes.

The effect of 2011 off Tohoku Earthquake and Tsunami is not limited to the shallow sea. Noguchi *et al.*<sup>[28]</sup> reported the occurrence of highly turbid bottom water in the forearc slope region. Arai *et al.*<sup>[5]</sup> reported the flow of turbidity currents originating from the tsunamis at about a depth of 1000 m in the forearc slope region, and Ikehara *et al.*<sup>[29]</sup> showed that

an event deposit was present in wide-ranging submarine surfaces down to a depth of 5500 m from the outer shelf to the mid slope. McHugh *et al.*<sup>[9]</sup> reported that there was wide distribution of an event layer originating from the 2011 earthquake and tsunamis on the flat area at a depth of 5000–6000 m called the mid-slope terrace off Sanriku. Moreover, Oguri *et al.*<sup>[4]</sup> reported suspension at the bottom water in the Japan Trench, and reported the deposition of event deposits containing cesium 137 and excess lead 210. Ikehara *et al.*<sup>[7]</sup> confirmed similar deposits from several points nearby, and concluded from its deposition structure that it was a deposit of fine-grained turbidites.

The suspension phenomena of the bottom water during an earthquake reported for the 2011 earthquake by Noguchi et al.<sup>[28]</sup> and Oguri et al.<sup>[4]</sup> have been observed in other earthquakes. Ashi et al.<sup>[30]</sup> reported the suspension of the bottom water in the sea basin on the landside of the Nankai Trough slope after the 2004 off the Kii Peninsula earthquake (M7.4), and the presence of muddy water with extremely high concentration of suspended material immediately above the sea floor, and the thickness of this material was estimated to be about 2 m. This result raises speculation of a process in which a pond of mud water (mud pond) with a thickness of about 2 m might have been formed in the depression of the sea floor, and mud might have accumulated to form the earthquake event deposit. Moreover, Ashi et al.[31] postulated that such a mud pond can be formed by resuspension of a few cm of unconsolidated deposits that cover a slope, estimated from the surface area of the depression, the thickness of the mud pond, and the surface area of the slope that could supply the mud to the depression. The presence of such a



### Fig. 2 Sediment formed by the tsunamis in the 2011 off Tohoku Earthquake; collected off Sendai (A: soft X-ray radiograph, B: X-ray CT image of upper layer, C: X-ray CT image of lower layer)

The sediment is composed of two units. The upper unit (upper portion above the arrows) is composed of fine-grained turbidites that become finer in the upper part. This is composed of two turbidite layers that are separated by an internal erosion surface (a surface formed in a series of sediment that cuts the lower sediment; shown in black dotted line). The upper and lower turbidites also become finer on upward (corresponding to the grain-size change from sand to mud, the change is from white to black in Fig. 2A, and from orange-yellow to blue in Fig. 2B). The lower unit is composed of hemipelagic mud that shows abundant disturbance by benthic organisms, but the deformation structure (white dotted line) caused by seismic movement can be seen at the upper part of the lower unit. Although the X-ray CT device allows obtaining 3D data nondestructively and the deformation structure due to seismic movement can be observed easily, the resolution is rather low for observing details of the deposition structure.

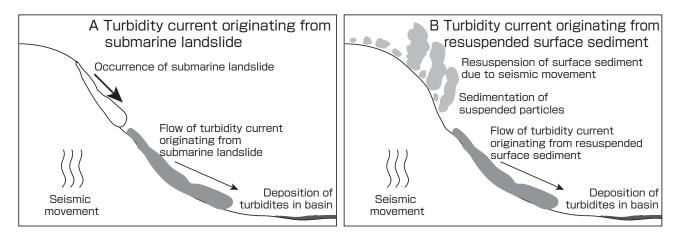
process, in which turbidites are formed by the generation of turbidity currents as unconsolidated sediment in the uppermost surface layer of the sea floor is resuspended by seismic movement (Fig. 3B), has been suggested from the sediment color measurement results and organic matter contents of the turbidites of the lakes in Chile,<sup>[32]</sup> and the radioactivity measurement on the landside slope of the Japan Trench.<sup>[9]</sup> Concerning actual occurrence of resuspension of marine sediment by seismic movement, Oguri et al.<sup>[33]</sup> conducted continuous submarine observations off Sanriku, and observed the resuspension and the deposition of surface sediments during an aftershock (M7.3) of the 2011 off Tohoku Earthquake. Perhaps the deposition of turbidites at the tip of submarine landslides and submarine debris avalanches, as shown in Fig. 3A, along with the presence of horseshoe landforms that seemed to be submarine landslides as seen on the bathymetric maps, might have provided an assumption to the researchers that the collapse of submarine slopes during an earthquake is essential in the formation of seismo-turbidites. However, in the resuspension process of surface sediments, the occurrence of large submarine landslides is not necessary for the formation of seismoturbidites. Moreover, if the sedimentation rate is sufficiently large against the frequency of earthquake occurrence, sediment that comprises the turbidites that is resuspended during earthquakes is stored on the slopes during intervals of earthquakes, and it will be possible for turbidites to be formed at each earthquake. For example, in a place where the sedimentation rate at the landside slope is 50 cm/1000 year, and the average earthquake occurrence interval is 200 years, 10 cm of sediment is formed on the slopes between earthquakes, and surface sediment of several cm can be supplied sufficiently enough to form turbidites in the next earthquake. On the other hand, in a place where the sedimentation rate is 10 cm/1000 years, there will be 2 cm of sediment deposited between earthquakes, and when the deposits are resuspended during an earthquake, slopes are

eroded and old sediment with low water content is exposed. Therefore, it is estimated that turbidites are not formed at each earthquake at such locations.

Deformation structures by seismic motion is another seismic record in marine sediments. There are several structures that are reported as deformation structures by seismic movement.<sup>[34]</sup> Sakaguchi et al.<sup>[35]</sup> reported a structure where surface sediment breaks in a breccia pattern due to seismic movements at the topmost part of a drill core from the landside slopes of the Nankai Trough, and stated it was formed in the 1944 Tonankai Earthquake, from the measurement of excess lead 210. Ikehara et al.<sup>[6]</sup> reported a linear structure arranged vertically (Fig. 2C) at the outer shelf off Sendai, and showed that it had a different size and interval compared to the vein structure observed in the mudstone of Kazusa Group that was formed by seismic movement,<sup>[36][37]</sup> but had the same form and length/interval ratio. Moreover, since this deformation structure was covered by event deposits formed by the 2011 tsunami, it was thought to be formed by the seismic movement of 2011. Such estimation of earthquake recurrence history using sediment with deformation structures caused by seismic movements has been used to study the lake sediments in Switzerland.<sup>[34]</sup>

# 3 Status of investigation of earthquake recurrence history using seismo-turbidites

Investigation of earthquake recurrence history using turbidites that are intercalated in the sediment of sea and lake floors are being conducted around the world.<sup>[7][18][34][38]-[40]</sup> Many papers have been published in the last few years. Other than the aforementioned disturbance and resuspension by seismic movements and tsunamis, turbidity currents can be caused by surge currents during storms, hyperpycnal flows that flow out of rivers during flooding, liquefaction of sediment due to repeated load of strong waves during



### Fig. 3 Two deposition models of seismo-turbidites

(A) Origin from submarine landslide in which a submarine slope failure occurs due to seismic movement, and (B) origin from resuspended surface sediments where the unconsolidated surface sediments are agitated due to seismic movement.

storms, increased pore pressure due to rapid deposition of deposits, groundwater outflow on the sea floor, volcanic eruptions, fall of extraterrestrial objects, and others.<sup>[39][41][42]</sup> Therefore, it is important to identify the cause of formation of the turbidites found in sediments, but it is not easy to discern the cause of formation from the characteristics of the turbidites themselves. In the turbidity currents originating from rivers and shallow sea regions, particles originating from land or shallow sea can be expected, and these can be used to identify the cause of formation.<sup>[43]</sup> For example, if an underwater delta front at a river mouth collapses during an earthquake, the particles that flow out have almost the same composition as the river sediments, and it will be difficult to identify the cause of formation from grain composition only. Therefore, in current practice, obtained turbidites are often considered seismic, based on the geographical and sedimentological settings that receive little effect of the shallow sea, such as if the sampling point of sediment is distant from a submarine canyon that continues from a river, or is within an independent depression without any submarine canyons. Therefore, depression formed along a fault movement on a landward slope of a trench (slope basin) is considered as one of the optimal places for studying earthquake occurrence history using turbidites.<sup>[44]</sup> Also, the formation of turbidites during an earthquake of a certain magnitude or larger and their accumulation as a deposit record are important factors. It is hardly known what kind

of earthquake event deposit was formed by an earthquake of what magnitude in a particular place. However, as mentioned earlier, in places where turbidites are formed by resuspension and redeposition of surface sediments, it is necessary for sufficiently larger amount of sediment to deposit on the slopes between earthquakes, compared to the amount of sediment that become resuspended. Also, for an earthquake event deposit to escape from the disturbance by benthic organism activities and the physical erosion of the sea floor so it could be used accurately as deposit records, it should have plenty of volume that accumulates between earthquakes, and should be away from sea floor surfaces that are easily subject to erosion and benthic organism disturbance. In practice, it is extremely important to select such locations from submarine topography and sedimentation to gather high quality samples for analysis.<sup>[39][44][45]</sup> Not all samples collected can be used to investigate earthquake recurrence history.

In the estimation of earthquake recurrence history using earthquake event deposits such as turbidites, the samples collected from appropriate places are used to identify the horizon of earthquake event layers intercalated in the samples, and the age of past earthquake occurrences are determined by studying the depositional ages of earthquake event deposits (Fig. 4).<sup>[39]</sup> Also, the epicenter and the size of rupture areas are estimated from the spatial distribution of earthquake event deposits formed at the same time. For the identification

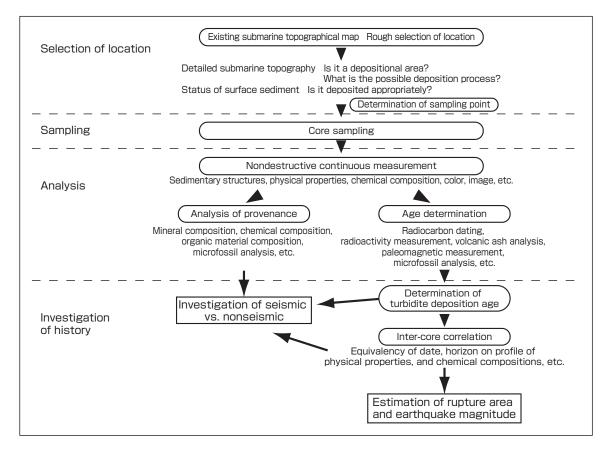


Fig. 4 Method for estimating earthquake recurrence history using seismo-turbidites

of the horizon of the event layer, sedimentary structures that could be observed by the naked eye, X-ray images, or X-ray CT images, and change in sediment color, grain-size, bulk density, susceptibility, mineral and chemical compositions, and others are used (Fig. 4).<sup>[39]</sup> In some cases, microfossils and organic compositions in the deposits may be used as indices. The identification of the earthquake event deposit by combining these techniques is essential.

The most common method for determining the depositional age of marine sediments is the radiocarbon dating method using planktonic foraminifera tests. Other methods include identification and correlation of obtained volcanic ash with age-known volcanic ash, correlation with geomagnetic paleosecular variation and paleomagnetic intensity variation curves, microfossil biostratigraphy, oxygen isotope stratigraphy of planktonic and benthic foraminifera, and radiocarbon dating using organic matters in the sediments. Of these, the method with the highest dating accuracy is the volcanic ash of historic eruption for which the eruption date has been determined. The radiocarbon dating of planktonic foraminifera that is most regularly used has errors of several dozen years (up to several thousand years ago) to several hundred years (several 10,000 years ago). Recently, there are attempts to calculate the event dates by considering the error using the Bayesian statistics for the probability distribution of individual date values, but needless to say, it is better to have as many datings as possible to obtain the reliable event date.

For the more reliable correlation of event layers between samples, it is desirable to compare from a position on the continuous data (profile) such as the geomagnetic paleosecular variation curve, color value, physical property value, or element concentration, while referencing radiocarbon dating and using the absolute isochronous surface such as mutually correlative volcanic ash as the standard. It is also preferable to obtain numerical data with high resolution as much as possible. To achieve this, nondestructive measurement is advantageous. Since there are many types of nondestructive analyzing devices, events can be compared by combining the profiles of multiple items with high resolution, and by adding many dates, it is expected to increase the accuracy of dating and earthquake event correlation.

If it becomes possible to compare earthquake events among the samples, it becomes possible to estimate the spatial distribution of turbidite deposition of a particular earthquake. From the results of submarine surveys conducted immediately after an earthquake, as the spatial distribution of seismo-turbidites is often limited to the earthquake rupture area and its proximity,<sup>[17][18]</sup> it is expected that estimation of earthquake magnitude and location of the rupture area will become possible from the distribution of seismo-turbidites. Some seismic movement deformation structures are thought to be related to the magnitude of seismic movement.<sup>[46]</sup> There are attempts to estimate the earthquake magnitude from the spatial distribution of seismic movement deformation structures and seismo-turbidites.<sup>[34]</sup> If this becomes possible, the estimations of earthquake occurrence location and magnitude become possible as well as the time of earthquake occurrence. But in the current situation, the establishment of an accurate comparison method for seismic deposits is the major issue.

# 4 Importance of background data to better understand the phenomena that occur on the sea floor during earthquakes

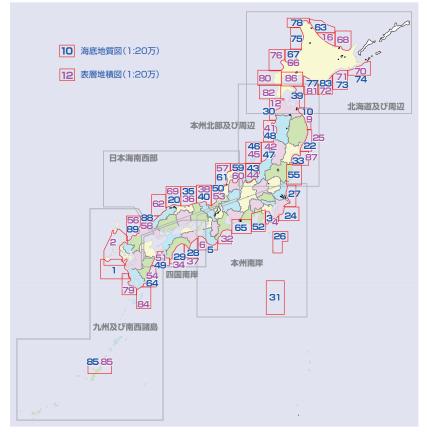
We have already shown that in the survey after the 2011 off Tohoku Earthquake, resuspension, reworking, and redeposition of the sediments occur on the sea floor due to earthquakes or tsunamis. However, it is rather difficult to clearly show that such sediment was formed by the earthquake or tsunamis of 2011. It is also difficult to quantitatively measure the amount of transported sediment on the sea floor. One of the reasons is because most of the surveys of marine sediments are done after geological or climatic events such as giant earthquakes and tsunamis or floods, while very few surveys are conducted to observe the sea floor during normal times. Data before an event that can be compared with survey results after an event is important for quantitative understanding of the phenomena that occur on the sea floor during an event.

AIST has been engaging in surveys for marine geological maps of the seas around Japan since the 1970s, and it has been working on sedimentological maps (Fig. 5). A sedimentological map is a geoscience map that presents the transport and deposition processes of sediments over the past tens of thousands of years, rather than a bottom sediment map that merely shows the distribution of sediments. It adds the analysis results of submarine photographs and subbottom profiling records to the analysis of grain size and composition of marine sediments collected from sampling points/ arranged in lattice at intervals of 4-7 nautical miles (7.4–13 km), although the intervals may change according to the sea area of the map segment, depth, and duration of survey cruise. For one sheet of sedimentological map that is compiled at the scale of 1:200,000, about 100 mud sampling points are set. While sediment sampling in an ordinary marine geological survey is conducted for a specific objective, the sediment sampling for sedimentological maps is done by arranging the sampling points at even intervals of the map area, to obtain even and uniform information of marine sediment. If necessary, samples are collected to clarify a specific objective, and supplementary sampling is conducted. AIST is the only organization in Japan that systematically collects marine sediment information.

The data for sedimentological maps can be used to evaluate

effects on the sea floor in events such as earthquakes and tsunamis or floods. In the case of the 2011 off Tohoku Earthquake and Tsunami, the sea floor photos obtained by AIST for the sedimentological map from Sanriku to off Sendai Bay were used to analyze the changes in benthic organisms before and after the earthquake and tsunamis, and it was shown that there were changes in the distribution of brittle star (Ophiurida), the major benthic organism of this area.<sup>[47]</sup> Also, the comparison of sediments in the Sendai Bay before and after the 2011 off Tohoku Earthquake and Tsunami showed that the disturbance and redeposition of sediment occurred throughout the Sendai Bay due to tsunamis.<sup>[48]</sup> Considering the later analysis results, maximum of about 10 cm of surface sediment was resuspended, and the state of sediment suspension continued for several days or more. Comparison of the surface sediment before and after an earthquake and tsunami clearly indicates what happened or did not happen during the events. The accumulation of these facts is expected to be useful for estimating what may happen and where on the sea floor it may happen during future giant earthquakes and tsunamis.

Comprehensively gathered sea floor sediment samples and information can be used for estimating the origin of turbidity currents arising from earthquakes and tsunamis. To know where a turbidity current occurred due to the disturbance on the sea floor by an earthquake and tsunami, how it flowed, and where it formed turbidites is important in accurately investigating earthquake recurrence history using turbidites. But at this point, there are few cases where the origins and flow routes are accurately estimated. In the process of flowing, a turbidity current deposits part of sedimentary particles contained in the turbidity current, while on the other hand, particles of surface sediments are entrained in the current at the head of the flow.<sup>[11]</sup> Therefore, not all particles that comprise turbidites indicate the origin of the current. On the other hand, it has not lost all of the particles of the place of origin. For example, this is shown in the fact that the turbidites at the bottom of the Nankai Trough off Shikoku contain particles of the Fuji River that is over 600 km away that is thought to be their origin.<sup>[49]</sup> Therefore, information on the sediment samples and their compositions at the origin or along the flow route is important in estimating the origin and flow route of a turbidity current. For the surface sediment samples collected comprehensively to create the sedimentological maps, the grain size distribution, particle composition, and chemical composition are analyzed and published.<sup>[50]</sup> For the recent estimation of particle sources,



#### Fig. 5 Publication of marine geological maps by AIST

Frames containing red numerals are areas where 1:200,000 sedimentological maps have been published. Frames containing blue numerals are areas with published 1:200,000 marine geological maps, and grey frames are places with published 1:1,000,000 marine geological maps. From the homepage of Geological Survey of Japan, AIST.

other than the major element composition, various minor and trace elements and isotope ratio are used.<sup>[51]</sup> The information of the geographical distribution of such chemical composition before earthquakes or tsunamis is expected to be useful in estimating the origin and flow route of turbidity currents.

### **5** Summary and future prospects

Deep sea sediments are less likely to be affected by sea level fluctuations, compared to the tsunami deposits on land that are affected by the changes in coastal landforms, including the changes of position of coastlines due to supply of sediment from the land as well as the rising sea level after the last glacial period. Marine sediments are expected to serve as recording media of the history of recurrence of earthquakes and tsunamis over long periods of time.<sup>[50]</sup> Therefore, attempts are made around the world to decode earthquake recurrence history using submarine earthquake event deposits. However, it is not easy to understand the situation of the deep sea floor where one must collect samples from depth of several thousand meters or more, and to obtain high quality samples from appropriate locations. As mentioned earlier, it is necessary to select the appropriate place from the geological cross-section and submarine topographic data with high accuracy and high resolution. AIST's marine geological map and the data and samples on which they are based are the basic data for selecting such locations. Therefore, it is necessary to further conduct the surveys for compiling marine geological maps and to quickly publish the results. Also, surveys of the main four islands of Japan have been completed for the marine geological maps, but the survey regions (Fig. 5) tend to be on the landside compared to the sites of earthquake occurrence around Japan. Preparation of basic marine geological information for the offshore area in which giant earthquakes are expected to occur is necessary in the future.

After high quality samples are collected, it is necessary to conduct analysis at high resolution as much as possible. As mentioned earlier, the identification of event layers is possible from quick and high-resolution data, by combining several nondestructive measurement devices that are being developed recently. Such quick analysis enables analysis of several samples, and contributes to the construction of more reliable data. It is desirable to build such an analysis system at AIST. On the other hand, an event deposit that is comprised from different deposition processes than ordinary sediment is thought to have different grain size, grain composition, and structure compared to ordinary sediments. Such a difference probably makes the appearance of the sediment different. It is important to nurture "eyes to read sediment" to understand the difference in appearance, without relying solely on analysis data. The "eyes to read sediment" is nurtured by looking consciously at various and numerous sediment samples. Also, the experience increases

by repeatedly observing samples and comparing them with abnormal values and change patterns in nondestructive measurement. It is important to constantly be aware of what creates the differences and changes in the sediment, not just to observe the event deposit through nondestructive measurement data. Moreover, in the interpretation of the nondestructive measurement results, there were many instances in which knowledge gained in places that may be irrelevant to earthquake history research such as the paleoenvironmental change was useful. To nurture the "eyes to read sediment," the effort to widen the horizon, not just gain experience in one thing, is important. We think it is particularly important for young researchers to gain as much experience as possible.

In Japan, which is an earthquake-prone nation located in the plate boundaries of the West Pacific, the knowledge of recurrence history of past earthquakes and tsunamis is basic information for safe and secure living. We hope to collect and analyze marine geological information to contribute to the safety and security of society.

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for obtaining earthquake recurrence history from seismoturbidites and disturbance of sea floor sediment during earthquakes.

### **Discussions with Reviewers**

#### 1 Overall

#### Comment (Masahiko Makino, AIST)

This paper discusses new scientific usefulness that may contribute to earthquake disaster prevention through clarification of the history of occurrence of past earthquakes and tsunamis, by capturing geological evidences in the submarine earthquake and tsunami deposits. The authors have yielded many results through active research by collaborating with other institutions since the 2011 off the Pacific coast of Tohoku Earthquake.

Specifically, the paper discusses the following topics: 1) the process of turbidite formation, 2) the deposits collected in the Sendai Bay that were formed by the tsunamis in the 2011 off Tohoku Earthquake, and 3) the estimation method for earthquake occurrence history using turbidites. It also states that the organization of intellectual infrastructure of marine geological information is important, because high-precision and high-resolution submarine topographic data and geological cross-section records are necessary to advance this study further.

This research is important from the perspective of earthquake disaster prevention, and I think it is appropriate for publication in *Synthesiology*.

#### Answer (Ken Ikehara)

In the 2011 off the Pacific coast of Tohoku Earthquake, the earthquake and tsunamis caused major damage in wide-ranging areas, as well as in the Tohoku region. The research of earthquake recurrence history using marine sediment presented in this paper had been conducted before the earthquake and tsunamis in 2011, but I think the advancement of research after 2011 is remarkable. Details of what happened on the sea floor during the earthquake and tsunamis were observed and recorded. Although there is much that is still unknown, I hope we can continue our survey and research, to contribute to society through our marine geological research.

#### Comment (Masanori Goto, AIST)

This paper is about the strategy for clarifying the history of giant earthquakes and tsunamis by analyzing the turbidites in marine sediment, while considering contributing to safe and secure living. It addresses the importance of regular data collection and the role of AIST as well as the state-of-the-art survey and analysis. It is arranged so that it is useful for readers outside the field, and I think it has value to be published in *Synthesiology*.

#### Answer (Ken Ikehara)

I wrote with contribution of survey and research to society in mind. I also wrote about how the assets of research conducted at AIST can be utilized, and how they should be utilized.

# 2 Explanation of turbidite

#### Comment (Masahiko Makino)

Is the water tank experiment for a turbidity current running down a slope in Fig. 1A cited from another paper? The text indicates Reference [11]. If it is a citation, please indicate that in the caption of the figure.

#### Answer (Ken Ikehara)

The photograph of the turbidity current produced in the water tank was taken by the principal author, and was not cited from another paper. The citation in the text is a part from another paper that explains the turbidity current. To avoid confusion, I moved the indicator of the citation to the appropriate place.

#### Comment (Masahiko Makino)

For Fig. 2, in this paper, the text explains the "two-story structure" of turbidites, while the figure caption refers to the "interior erosion plane." I think the formation process will be more easily understood if you explain the "interior erosion plane." **Answer (Ken Ikehara)** 

I added and revised the explanatory text based on your comment.

# 3 Technological issue

#### Comment (Masahiko Makino)

Were there any technological issues that were solved that allowed the dramatic advancement of the submarine earthquake and tsunami deposit research in recent years?

You mention it in Chapter 3, but can you organize it a bit more and make it more understandable?

#### Answer (Ken Ikehara)

As a technological factor, I think there is the advancement of nondestructive measurement for the physical and chemical compositions of core samples, as described in Chapter 5. This provides quick, high-resolution data.

In Chapter 3, we explain the research method, so for the point that you indicated, I added in Chapter 5 the fact that nondestructive measurement technology has been advancing recently. However, in practice, there are many researchers who do not look at the actual core, relying completely on measurement data. I believe "eyes to read sediment" are nurtured by looking and comparing data against the actual core, and I added this point to the paper.

#### Comment (Masahiko Makino)

In Fig. 4, you list the elemental technologies, but the arrows that join the elements are short, and I think it will be more understandable if you work a bit more on the overall layout. Can you enclose the technology clusters in blocks?

# Answer (Ken Ikehara)

I revised the figure based on your comment.

#### Question (Masanori Goto)

In the "Summary and future prospects," you address the importance of nurturing the "eyes to read sediment." You write that it is important to look with awareness and to gain experience, but can you be more specific about this? I think this is also applicable to researchers in other fields.

#### Answer (Ken Ikehara)

This is rather difficult to express in words, but I have experienced several times that things that seem to be irrelevant at a first glance are actually mutually related. It is, of course, important to gain experience, but I think it is more important to gain experience by tackling each topic seriously, and I added descriptions on this point to the text. Also, I think to understand properly how data change at what characteristic parts by comparing the results (data) of nondestructive measurement, which is advancing dramatically in recent years, and the actual material (core) is essential in nurturing the "eyes to read sediment." I also added this point to the text.

# High-accuracy endoscopic microscopy using a thin, 1.5 mm diameter probe with optical coherence tomography

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We developed an endoscopic microscopy system with 20 nm accuracy that affords inspection through narrow gaps using a thin, 1.5 mm diameter probe. Accuracy was improved using Optical Coherence Tomography (OCT). The frequency modulated light source is stabilized with closed control from self-interference measurement. The probe is driven by two miniature motors, which allow three-dimensional scanning of an internal surface. Imaging performance is 60 frames per second. The high accuracy with narrow clearance capabilities of this system reduces the need for machine overhauls, which affords trustworthy daily inspections and hence greater machine reliability.

Keywords: Endoscope, fiber scope, Optical Coherence Tomography (OCT), super resolution, internal diameter measurement

# **1** Introduction

How far will machining technology advance? In terms of accuracy, there is a famous roadmap known in the precision machining field as shown in Fig. 1.<sup>[1]</sup> Since atoms cannot be divided by machining process, 1 nm should be the fundamental limit which is close to the size of an atom, and nanotechnology is the way to approach that limit using various methods. In that sense, atomic manipulation<sup>[2]</sup> and molecular manipulation<sup>[3]</sup> using an atomic force microscope (AFM) have been realized, and in theory, further advancement cannot be expected. Yet, does the machining technology end there?

The ultra-high-accuracy machining technologies such

as AFM and focusing ion beams are not implemented immediately in personal-use 3D printing or computer numerical control (CNC) mills, and cost and time are not feasible. Recently, 3D printers and CNC mills have been in the spotlight as innovations in production technology, and these may change production technology from the basis. However, in fact, these were invented over 35 years ago. CNC was developed in 1952 as a project of the Massachusetts Institute of Technology,<sup>[4]</sup> and the 3D printer was invented by Hideo Kodama in 1980.<sup>[5][6]</sup> Incidentally, when Kodama presented a prototype and printed trial products, the engineers who were so engrossed in submicron level machining precision totally ignored him.<sup>[7]</sup> Even if technology is of the highest quality, the world will not change if it is useful only to a few. I think

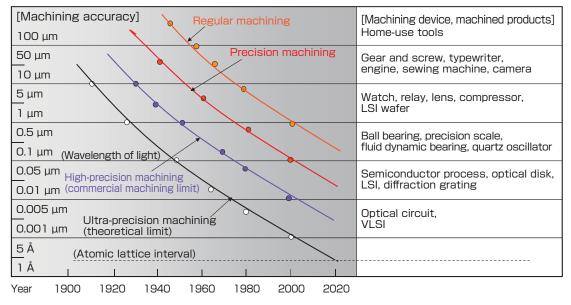


Fig. 1 Trend in machining accuracy

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evaluation as "innovation" and "novelty" can only be given to the technology that was limited to few researcher becomes widely available for personal use. In this sense, to reform the things that were invented into usable forms and to reduce cost are extremely important form of innovation. Including control technology and measurement technology, the majority of the manufacturing technologies have not reached atomic accuracy, and therefore, there is ample room for advancement.

We are currently looking at the technology for high-accuracy measurement of inner cylinders. Inner cylinders are not only used as combustion baskets of engines or power generator turbines, but are also components whose accuracy affects the energy efficiency of bearings and other parts. Despite this fact, since most of the technologies applicable to high-accuracy measurement have evolved from microscope technology, it is limited mostly for thin, flat samples, and is not suitable for inspection of inner side. For inner cylinder inspection, the current situation is going toward how to achieve high accuracy in conventional roundness measuring instruments. The current highest accuracy is about 0.05  $\mu$ m, and as long as it is an extension of roundness measuring instruments, expert craftsmanship is required for centering and leveling.

There are many limitations such as in accuracy, procedures, measurable sizes, and measurable shapes in the technology for measuring the inner diameter. They cause its low usability and application range. These equipment should be used not only for quality control during parts manufacturing, but be also used in routine inspection and load tests to determine the wear/tear and exchange period, and it is necessary that anyone can obtain highly accurate digital data that can be managed on a PC.

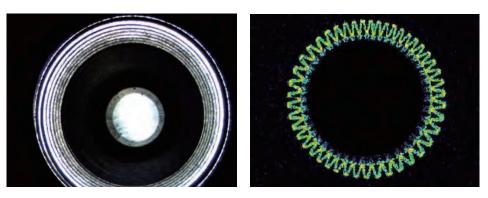
The endoscope would be suitable for this purpose. However, it usually provides still or motion images as seen in Fig. 2 left, which did in short-term diagnosis only. The digitized information would be more useful for the comparative analysis, as shown in Fig. 2 right. Since it is difficult to detect

minute shape variations of a subject with a photograph, comparison by digitized data is preferable. Regarding this point, rather than a conventional endoscope, we need a digital microscopic endoscope combining the functions of digital microscope and the endoscope-like probe that can enter small spaces.

In our research, the digital microscopic endoscopy has realized to perform high-accuracy measurement based on optical coherence tomography (OCT). Although optical interferometry used in OCT has been common in industrial surface measurement as white-light interferometry, Professor Tanno of Yamagata University and J. Fujimoto *et al.* of MIT almost simultaneously showed that this can be applicable to tomographic measurement of the fundus.<sup>[8]–[10]</sup> After these research, application to ophthalmology advanced rapidly, and the products with user-friendly interface are available from almost all ophthalmological device companies around the world. Innovative change was made in ophthalmological diagnosis when human retinal tomography became available.

Originally, OCT was configured using optical fibers, and it is suitable for the fabrication of microprobes, but its deployment in endoscopes has been slow. Endoscopes using OCT are used limitedly in the medical field, and vascular OCT with which blood vessels are imaged from inside has been drawing attention. On the other hand, when irradiating a vessel wall with measurement light emitted from a probe that is inserted into a vessel, blood interferes with the OCT optical path. Therefore, it is necessary to stop the blood flow by blocking the vessel for a short time using a balloon catheter, flush the blood by injecting physiological saline, and then observe the blood vessel wall. This requires more skill compared to the conventional intravascular ultrasound (IVUS), and the OCT method has not replaced the conventional method. An innovation that allows easy operation is necessary.

On the other hand, there is hardly any industrial application of OCT. While it will be discussed in the next chapter, the specifications required are different from medical application,



**Fig. 2 Examples of analog endoscope and digital endoscope** Although the samples are different, the photographs show an endoscopic photograph (left, conventional) and digitized data (right, our technology) of a tube interior.

and both optical and machine technologies that are different fields are necessary to fill in the gap. We took the approach of advancing the two technologies through collaboration between the Electronics and Photonics Research Institute, AIST (hereinafter, AIST) that specializes in optics, and Adamant Namiki Precision Jewel Co., Ltd. (hereinafter, Namiki) that specializes in precision machine technology. Namiki is number one in the world for the fabrication of micromotors that are key parts of probes. We set an extremely high goal value that was to measure the surface shape at 20 nm accuracy using a 1.5 mm diameter probe. Since such a measuring device did not exist previously, the field of application is yet unknown, but we expect there will be usage for inspection from small gaps and data accumulation, without disassembling machines, for generators, automobiles, aircraft, or any situations where rotation wear in machines must be addressed.

For example, domestic shipment of precision measurement devices was about 110 billion yen/year in 2016, according to data of the Japan Precision Measuring Instruments Manufacturers Association.<sup>[11]</sup> Considering that this research can be utilized in conventional roundness measurement instruments (16 billion yen/year), surface roughness testers (9 billion yen/year), 3D measuring machines (5 billion yen/year), and measurement of inner diameter of deep holes that cannot be measured by industrial CCD camera endoscopes, and assuming 30 % or more increase in the production of such instruments (about 30 billion yen), it is thought that a new market of about 10 billion yen/year scale will be formed.

# 2 Methods and Results

# 2.1 High-accuracy OCT by self-phase detection: 20 nm

Since OCT advanced as medical technology, it has already overcome the period of expensiveness which attends initial diffusion of technology, and currently, parts are available at a relatively reasonable price. Utilizing this merit, diffusion for industrial use is being prepared, but the issue of repeatability is the index that sets apart medical and industrial uses.

The performance required for medical OCT is to obtain a clear image of each layer of the retina, and the indices of a good image are mainly depth resolution and degree of penetration depth of light. One of the endoscope-type OCT used in medicine is vascular OCT.<sup>[12]</sup> On the other hand, the performance required for industrial OCT is the ability to accurately trace the surface of an object, and distance accuracy and repeatability are the main indices. Particularly, for industrial use, when the object is a metal surface, depth resolution and degree of depth are hardly necessary, and the direction of research is different. To address this issue, we used the following method.

The optical system we used was composed mainly of an optical fiber interferometer. It is a type called swept source OCT (SS-OCT) that uses a wavelength scanning light source. Figure 3 shows the schematic diagram. The light source is a swept source (Santec HSL-2100) whose wavelength range is 1240–1400 nm. The light source is divided into two fibers at a ratio of 95:5 at coupler 1, and the each beam was led to the Michelson interferometer to measure the distance and to the auto-interferometer for phase correction, respectively.

In the Michelson interferometer, the introduced light was separated into 95:5 by optical fiber coupler 2, for the reflective surface of the object to be measured and the reference reflective surface. The reflected lights from the object and the reference are coupled together at optical fiber coupler 3, and the interference signal is measured by a detector. Regarding this interferometer, the interference signal strengthens when the difference of the distance to the reflective surface of a sample and the distance to the reference reflective surface from the position where the beam is split at optical fiber

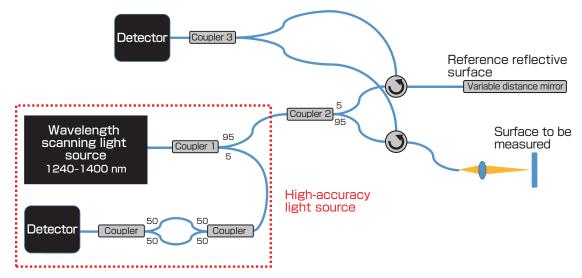


Fig. 3 Schematic diagram of high-accuracy OCT optical system

coupler 2 become the integral multiple of the light source wavelength. Therefore, the difference between each distance (optical-path difference) or each beam can be measured by scanning the light source wavelength. That is, when the sample surface is placed at a zero-path position (the same distance as the reference surface), the interference signal intensifies without dependency on the wavelength. When it departs from the zero-path position, fringes occur in the light source spectrum (spectral interference fringe patterns). The frequency of the spectral interference fringe becomes higher as the distance difference increases, you can calculate distance difference using the Fourier analysis.

Here, the distance difference is determined by the frequency of spectral interference fringes, and it is necessary that the frequency of spectral interference fringes be stable to conduct distance measurement with high accuracy. However, it is difficult to keep a constant speed of wavelength scanning. Although it may depend on the scanning mechanism of laser utilized, in general, the wavenumber variation often slows down at the beginning and end of scanning, and the light source wavenumber against time is nonlinear. This is shown in Fig. 4 left (before improvement). In this demonstration, a mirror is used as the sample, and the spectral interference of even intervals should be seen when the scanning speed is kept constant, but the spectral interference fringes were wide around 1240 nm at the beginning of wavelength scanning and around 1400 nm at the end of scanning, and they are narrow around 1320 nm at the center. In general, light source manufacturers correct this nonlinearity so it will become even with constant scanning speed by linking with detection software. Rough correction can be done using this method, but blurring at each scanning remains, and we conducted improvement of the correction by monitoring the scanning rate in real time by guiding part of the light source to the auto-interferometer. With the auto-interferometer, optical path difference between two divided beams is caused only by the difference of the length of two fibers, and therefore, if wavelength scanning is conducted at constant speed, spectral interference will occur at even intervals. If there is unevenness in wavelength scanning speed, which will be reflected in the fringe intervals of spectral interference, and intervals of sample detection signals can be corrected based on this information. According to this principle, even if there is unevenness in the light source, the constant scanning speed can be reproduced. As a result, as shown in Fig. 4 right (after improvement), constant measurement from start to finish of wavelength scanning has become possible, and stability of measurement positions increased. Figure 5 shows the result of the OCT position measurement before and after improvement. Before improvement, the standard deviation of measurement repeated 500 times was 380 nm, while after

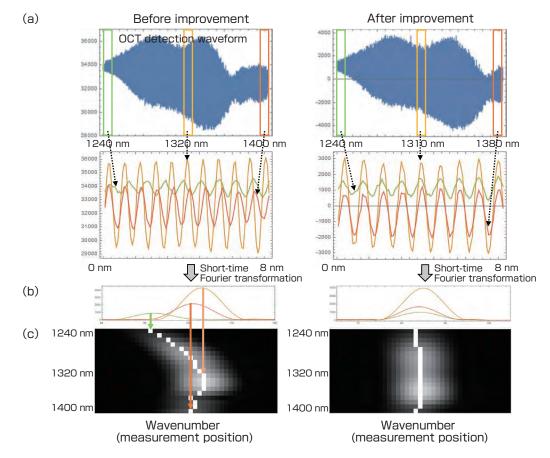


Fig. 4 Comparison of nonlinearity of interfering waveforms seen in a current light source (left), and the one after correction using auto-interferometer (right)

improvement, it was 22 nm, and improvement of about 17 times was obtained.

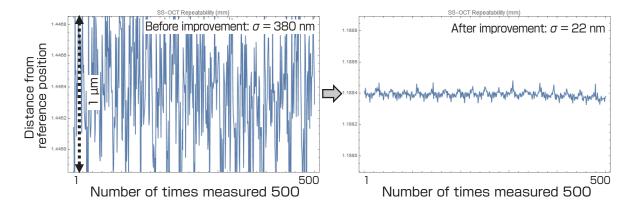
Applying this high-accuracy position measurement technology, shape measurement of a ten-yen coin was demonstrated as shown in Fig. 6 left. Roughness of the ten-yen coin was about 100  $\mu$ m, which was too large for verification, so the flat part of surface without unevenness was tested for the performance demonstration. Figure 6 right shows the result, whose height is magnified 100 times. Surface roughness was calculated as Ra = 0.31  $\mu$ m from the measured data. Since detailed surface shape measurement can be conducted, surface processing accuracy Ra, important in industrial measurement, can be calculated.

# 2.2 Rotating probe of 1.5 mm diameter [13]

Namiki developed a micromotor of 1.5 mm diameter for the first time in the world in 2005, and has succeeded in developing motors of 0.9–2.0 mm diameters, aiming for further miniaturization.

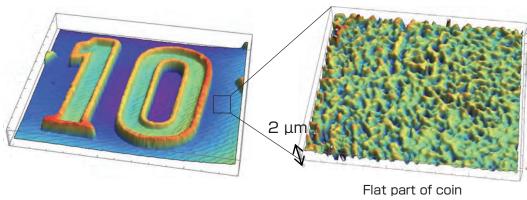
In this system, to compose a 3D scanning OCT probe of

1.5 mm diameter, we employed the micromotor of 1.5 mm and 0.9mm diameters for the two axes; tangential and axial scanning. As shown in Fig. 7a, the first micromotor installed at the tip of an OCT probe rotates the mirror using a motor of 1.5 mm diameter. The beam from the optical fiber becomes a side-illuminating beam that is bent 90 degrees, and this allows rotating scanning of 360 degrees. The second motor of 0.9 mm diameter has the role of changing the angle of the side-illuminating beam, and conducts scanning in the axial direction from front to rear. Specifically, the second motor rotates the optical fiber with a tip cut at a certain angle, and the direction of beams irradiated from the optical fiber is offcentered. By doing this, the beam position can be controlled to the upper or lower part of the mirror that is rotated by the first micromotor, and the angle of side-illuminating beams can be varied. The two motors can be synchronously rotated. For example, the first motor can be set to 3600 rpm and the second motor to 3540 rpm, and rotational phase is produced by giving slight rotational difference, and this enables helical scanning where the optical beam rotates at 3600 times per minute and engages in reciprocating motion of 60 times in the axial direction.



#### Fig. 5 Comparison of distance measurement accuracy before and after improvement

The vertical axis shows the position of the surface of the measured sample, and the horizontal axis shows the number of times measured. In measurement repeated 500 times, standard deviation improved from  $\sigma = 380$  nm to 22 nm.



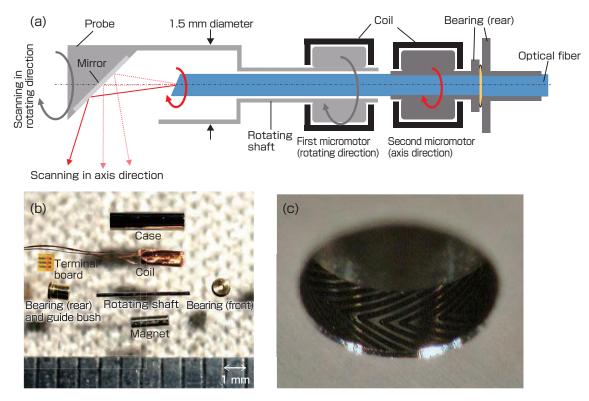
Surface roughness  $Ra = 0.31 (\mu m)$ 

Fig. 6 Measurement of height of a ten-yen coin by high-accuracy OCT (left: 10 mm square), and result of height measurement for the flat surface of a ten-yen coin (right: 1 mm square)

Since the second motor shown in Fig. 7a is extremely thin with 0.9 mm diameter, the brushless-coreless method was used. The parts used in this motor such as the rotating shaft, bearing, coil, and magnet are extremely fine as shown in Fig. 7b. The diameter of the rotating shaft is 0.2 mm, and the optical fiber passes through the center, requiring a hole of 0.125 mm diameter. For the coil, wire of 0.024 mm diameter is coiled at high density using a special winding machine, to maintain rotational torque. The bearing has internal diameter of 0.2 mm, and as shown in Fig. 7c, the herringbone dynamic pressure grooves are etched by lasers in the inner circumferential surface. When rotation starts, oil flows into the bearing along the groove, and the rotating shaft lifts

off the bearing as it is supported by oil pressure. This is called dynamic pressure bearing, where the rotation center stabilizes through noncontact rotation using the oil dynamic pressure that occurs by rotation, and accuracy in increased. Such extremely fine and almost artistic manufacturing technology increases the property of micromotors.

As shown in Fig. 8, this high-accuracy 1.5 mm endoscope was used for interior measurement of a hexagonal screw. The result is shown in Fig. 9. Stable digitization at 60 frames per sec was achieved.



### Fig. 7 Control mechanism at the tip of a micro-diameter probe

(a) Cross-sectional structure of rotating probe, (b) parts of second micromotor, and (c) herringbone dynamic pressure groove in the dynamic bearing

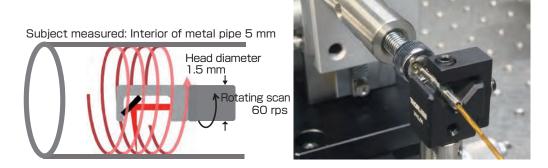


Fig. 8 Appearance of a rotating probe of 1.5 mm diameter (left), and measurement of a sample (head part of M6 screw) (right)

# 3 Discussion (breakthrough and impact)

With this device, a new endoscope is realized through the fusion of technology for noncontact shape measurement with 20 nm accuracy and technology for a full rotation probe of 1.5 mm diameter. Digitization of the interior at high accuracy through gaps may literally be a "gap" or niche industry, but if this becomes possible, daily inspections can be done without overhauls as shown in Fig. 10, and this may lead to the reduction of down time and daily management cost.

- (a) Nanometer level shape measurement using optical (noncontact) technology
- (b) Realization of a rotating probe that allows measurement from gaps (digitization, not just photographing)

By integrating the above two technologies, the inspection from gaps can be done easily and overhauls will not be necessary. An assumed target for this device is application to rotary driving devices such as, for example, jet engines that tend to wear quickly, routine management of generator turbines, and inner cylinder tests for small engines of

Result of measurement of

interior diameter 60 fps

rotating probe of 1.5 mm diameter

automobiles. In rotary driving devices such as engines, shapes of inner cylinders and polishing accuracy highly affects fuel consumption. While there are many devices that can measure the exterior, there are hardly any devices that can measure the inner cylinders. By managing the wear state and accretion through high-accuracy digitization, highaccuracy quality control can be conducted in a short time. We believe this is useful technology in R&D and production lines as well as routine management.

# 4 Future issues and prospects

In this paper, investigation was conducted using circular scanning in which the same track of the sample is repeatedly measured for accuracy assessment. As mentioned in the part about probe structure, this probe can conduct helical scan by combining axial direction scanning using the second motor, and therefore, measurement of 3D shapes can be done easily. However, to reconstruct 3D digital data, it is necessary to correct axial direction scanning by nanometer accuracy as shown in Fig. 11. We are considering working on this issue in the future. Using only rotational scanning, correct

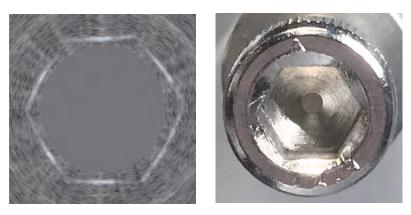


Fig. 9 Measurement of the interior diameter of a sample using a

Subject measured (work)

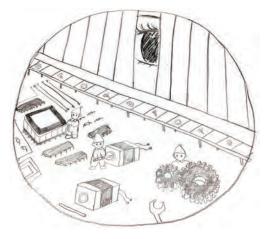


Fig. 10 Image of gap measurement

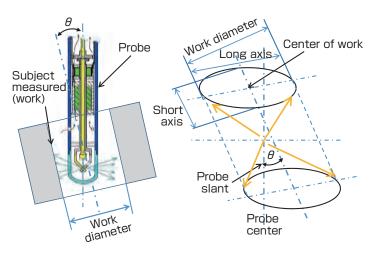


Fig. 11 Algorithm for tilt auto-correction

measurement cannot be taken since the image will be elliptic if the probe is tilted against the cylindrical axis when the probe is inserted in a cylindrical sample. If it is 3D shape, correct measurement can be taken no matter what the angle of insertion, and the usability will increase dramatically.

# **5** Conclusion

Part of this research was done with the grant from the Strategic Foundational Technology Improvement Support Operation. The screening interview at the start of a project is normally very strict, but in our project, the judges were impressed. Actually they were worried that our goal was set too high. They said, "Even if you are not able to achieve measurement accuracy of 20 nm, and the performance falls short by one digit, it is still sufficiently useful." I was quite grateful when the judges proposed how to lower the goal we should achieve. This was an understandable comment, and in fact, we debated over this, and the majority of the opinions of other optical measurement specialists was also that our goal setting was too strict.

There were three reasons for setting a high goal despite the concerns. First was the low accuracy of the existing devices for measuring the interior diameter of cylinders that was the main theme of this research, and as the judges said, this research would reach to top in the world even if we were one digit short of our goal. Second was that the machining technology level of Namiki and their desire to realize this technology were high, and improvement in optical technology in a short period was also expected. When we discussed how AIST could cooperate, Namiki already had a certain level of OCT-based technology that was totally self-made. Yet they were willing to take advice from us, and that eagerness seemed fresh to us. AIST has been conducting joint research with various companies, but Namiki's enthusiasm for realizing the technology was particularly outstanding, and that was impressive. We were even more surprised when we visited their Aomori plant. We met other members of the development group, and the cliché phrase "we work on development as one team" was truly put into practice. Starting with Plant Director Shibuya, all the staff had high degree of specialty, all the way to the people who were manufacturing the parts. They were truly professional. They correctly received instructions from others, but would not rely entirely on the given, and would work to realize something on their own. We were impressed. Third, there was consideration for the difference between research and manufacturing. In most cases in research, only one prototype device would be made, and accuracy meant "repeatability," or we had a feeling that accuracy was equal to the standard deviation of measurement values. However, since manufacturers suppose mass production, they emphasize standard errors, and this is important for quality control. If standard errors are considered, we can be less stringent.

Thus, research was started with a high goal, a strong will, and a little bit of leeway. As shown in this paper, the goal was achieved even when evaluation was done by repeatability without running away from our original goal. We are conducting technological development to enhance usability to create true innovation in the way we mentioned at the beginning of this paper.

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### Hiroshi YAMAZAKI

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From 2012, started working on the development of precision measurement instrument that applies fine diameter OCT probe through integration and application of optical parts machining and technologies of micromotor machining and assembly that are unique technologies of the company.

## **Discussions with Reviewers**

#### 1 Overall

#### Comment (Motoyuki Akamatsu and Ken'ichi Fujii, AIST)

This paper is about an innovative development where Optical Coherence Tomography (OCT) and very small motors were combined, thus allowing the insertion of a microscopic endoscope into narrow gaps of 1.5 mm diameter, and making possible the assessment of the interior of a cylinder at 20 mm precision. The OCT started as a technology using optical fibers, and since it is highly compatible with optical fiber endoscope devices, an endoscopic microscope with the world's highest precision was realized by achieving high-accuracy for OCT by developing autophase detection technology, and building this into a probe driven by the world's top class micromotor. In addition to shortening the time required for quality control inspection, routine inspection of the interior of devices becomes possible without overhauls. Medical applications such as vascular OCT to check the interior wall of blood vessels is also considered, and it is a measurement assessment technology with future expandability.

#### 2 R&D of core technology Question (Ken'ichi Fujii)

As a factor that allowed successful and dramatic increase in accuracy of interior distance (unevenness) measurement to 22 nm, which was conventionally about 380 nm, you mention that part of the light source was guided to the auto-interferometer and this allowed real time monitoring of the wavelength scanning speed. You also mention that this led to the stability of the frequency of spectral interference fringes and increased the stability of distance measurement. Specifically, did you conduct distance measurement using the phase-shifting method? I think the readers will deepen their understanding if you provide more details on the optical phase measurement methods and also explain the principles of surface shape measurement.

#### Answer (Hiromitsu Furukawa)

Specifically, the wavelength scanning speed of a light source was monitored using an auto-interferometer, and the detected light was corrected as if the scanning speed was kept constant. I made changes to Fig. 4 so the readers could understand easily, and I added a text to explain the technology based on this figure.

#### 3 Application of the endoscopic digital microscope Comment (Ken'ichi Fujii)

You give application such as to routine inspections of jet engines and turbines utilizing the advantage of a probe being able to be inserted into gaps of 1.5 mm. I think with further downsizing, you can apply this technology to the observation of internal walls of blood vessels and living tissues. If you are considering application to uses other than machine inspection, please elaborate.

#### Answer (Hiromitsu Furukawa)

As you indicated, medical application was realized earlier, and I described vascular OCT as an example of such application. As you commented, we can also apply this technology to observation of living tissues. Namiki's motor has been cited in the papers of vascular OCT, and I think we must be prepared for progress in that field.

However, although industrial application is also important, it is not done as much. That is because industrial application requires higher specs than for medical, yet the cost must be kept low. Therefore, in this research, we worked on increasing the performance that differ from that of vascular OCT, to create an OCT endoscope suitable for industrial application.

# 4 Control mechanism of the micro-diameter probe tip Question (Ken'ichi Fujii)

Figure 7 explains the micro-diameter probe using motors of 0.9 mm and 1.5 mm diameters that were developed by Namiki. I think you should provide explanation of the principles and mechanisms of such micromotors, to promote application of the motor itself.

Answer (Hiromitsu Furukawa)

I added the structure photo of the micromotor (Figs. 7b and 7c) to Fig. 7, and added a text to explain the mechanisms of motors and dynamic bearings.

#### 5 Integration of technology Question (Motoyuki Akamatsu)

I can see that you realized high-accuracy internal diameter measurement by combining OCT and micromotor-driven probe technologies, but why did you start working on this technology in the first place? Were you planning to use OCT technology from the beginning? Were you looking for application of the OCT technology? There might have been several factors, but what was the actual story? I think such a story will be beneficial for the readers.

#### Answer (Hiromitsu Furukawa)

When Namiki visited AIST, they already had the basic technology for OCT and I think they had enough technology to complete the device on their own. Normally, a company will think about completing the product on its own, but they consulted AIST, and while I felt honored to be consulted, I also wondered why they consulted us. But when I visited the Aomori Plant, my query faded, and I added some description of this process in "Chapter 5 Conclusion." Although it is not exactly an encounter, I hope it will be useful to the readers to see a corporate culture that truly promotes new ideas in manufacturing.

# Materials recycling technology for recovering rare earth fluorescent powder from fluorescent lamp sludge

# - Pioneering near-future resource circulation-

# Tatsuya Oki<sup>1\*</sup>, Tomoko Akai<sup>2</sup> and Masaru Yamashita<sup>2</sup>

[Translation from Synthesiology, Vol.11, No.1, p.33-45 (2018)]

A materials recycling technology to recover green phosphor, including terbium, which is a heavy rare earth, from fluorescent powder in waste lamp sludge was realized by collaborating with researchers who specialize in materials and powder sorting. There are few cases worldwide in which materials circulation from post-consumer waste has been established in loops further inside than horizontal recycling. This is a recycling system which is described as an ideal circulation system for the near future in Europe's circular economy (CE)/resource efficiency (RE) policies, etc. This study is a successful example of Japan leading the world in urban mine development to establish a resource circulation system of various waste products, and becoming a pioneer in near-future resource recycling.

Keywords: Rare earth, fluorescent powder, high gradient magnetic separation, magnetic matrix, terbium, urban mine

### **1** Introduction

Japan relies on imports for most of metal resource materials. It is the world's largest importer of rare metals that are essential in manufacturing high-tech devices, and one may say that the manufacturing industry exists through the import of rare metal resources. The problems of rare metal supplies are that their production control is difficult as many metals are by-products, they invite oligopoly since the consumption volume is low, and they are influenced easily by international affairs. Compared to base metals that are heading toward gradual depletion, rare metals are susceptible to man-made factors such as the affairs of the producing countries, and it is difficult to forecast which rare metals will have the supplies cut off,.

The rare earths (17 elements) are types of rare metals, and their market is dominated by China. The "rare earth crisis" is still fresh in our memory, as the domestic industries were hit hard, although temporarily, by the trade embargo against Japan and the following restrictions of export volume. Rare earth resources exist throughout the world including the USA and Australia, and recently, submarine deposits in the Japanese exclusive economic zone (EEZ) was in the news. The reason why China captured the current market is because they possess extremely good mines. One factor is that the products from the Chinese mines hardly contain any radioactive impurities seen in other rare earth mines. Therefore, only simple processing is necessary for production. Another factor is that they have heavy rare earth products such as dysprosium and terbium that are used as magnetic and fluorescent materials. Particularly, in the world movement toward low carbonization, the heavy rare earth resources that are necessary for the manufacture of highperformance motors are attracting high attention.

This research was started before the rare earth crisis became serious. Around 2005, the price of rare earth was gradually increasing, and the urban mine resources in Japan that were practically untouched suddenly gained attention. At the time, the authors were conducting research and development of physical sorting technology of magnets and capacitors,<sup>[1]</sup> while the objective of this research was to concentrate and recover green phosphor LAP that contains terbium abundantly from waste fluorescent lamps. The fluorescent lamps are roughly divided into white light types and threewavelength (rare earth) types. Three types of fluorescent powder corresponding to RGB are used in the threewavelength type lamps. The topics of this research are as follows: to remove white fluorescent powder, glass, and other impurities from the mixed sludge of white and threewavelength waste fluorescent powders, to concentrate the green (G) LAP, and then to reuse it as fluorescent powder material.

Traditionally, fluorescent powder sorting was studied at universities, and sorting using particle density and surface hydrophobicity/hydrophilicity was attempted. However, it was difficult to separate the white and three-wavelength types, and no practical application was achieved. A hint for success of this research was found when the authors (Akai and Yamashita) saw that LAP selectively attaches to the magnet when it is placed under extremely strong magnetic force, although it seems to be unresponsive to magnets. To

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utilize this principle in industry, a high-gradient magnetic separator was improved and made into a device to achieve highly concentrated LAP (Oki). In this research, a separation system was developed and brought to practical realization through the collaboration of researchers specializing in materials and sorting. The following is an account of the road to practical realization particularly from the viewpoint of separator device development.

# 2 Building up to the development of a separator

### 2.1 Research prior to the selection of topics (2005–2006)

The author (Oki) started investigation of recycling technology for waste products that contain rare metals from the end of 2005. In a survey project of Japan Oil, Gas and Metals National Corporation (JOGMEC), advanced recycling method was investigated for small fuel cells, automobile catalysts, neodymium magnets, and fluorescent lamps. At that time in the recycling plants, automation technology for cutting the two ends of straight fluorescent lamps, recovering glass and aluminum, and then processing the waste fluorescent powder including mercury had been established. The transition from incandescent light bulbs to compact fluorescent lamps was occurring in Japan at the time, and it was necessary to establish a recycling method. Therefore, in the succeeding project, selective crushing and separation methods for fluorescent powder of compact fluorescent lamps were studied. However, compared to straight tubes, there were numerous types of compact or bulb type fluorescent lamps, and there was no clear idea for developing a comprehensive processing technology.

On the other hand, a researcher at AIST was looking for application of magnetic levitation in the bioscience field, and was referred to the authors (Akai and Yamashita) to see whether it could be used in this research. At the time, we were conducting glass research, and it seemed that using magnetic levitation by superconductive magnets (equivalent to a suspension magnetic separator by the wet method) for recycling waste glass would not be cost effective in terms of resource value. Therefore, we suggested using this technology for waste fluorescent powder because it was more valuable, and started joint investigation. At the same time, the Rare Metal Task Force at AIST was working on a method for recovering terbium from LAP by a vitrification method,<sup>[2]</sup> but this also seemed to be difficult cost wise. An engineer at a major lamp company told us that there was a social demand for technology to recover reusable LAP from waste fluorescent powder since LAP deteriorated very little.

### 2.2 Identification of topics and start of R&D (2007–2011)

In 2007, the authors (Akai and Yamashita) conducted fluorescent powder separation tests by magnetic levitation, and confirmed that LAP could be levitated selectively by adding an appropriate surfactant. Magnetic levitation tests for various fluorescent powders were conducted, and a separation method of fluorescent powder by magnetic levitation<sup>[3]</sup> was established. At this time, the price of rare earths started to increase, and the lamp manufacturers became highly interested in this technology, and we started seriously preparing for a project proposal. During this preparation stage, there was a consultation with an author (Oki) who specialized in sorting technology and had been studying recycling technology of fluorescent lamps. Oki responded that realizability was higher for a high-gradient magnetic separator rather than magnetic levitation, and therefore, steps were taken towards the development of a high-gradient magnetic separator.

In 2009, this research was selected as the "Rare Metal Substitute Materials Development Project" of the New Energy and Industrial Technology Development Organization (NEDO), and the development of recycling technology using high-gradient magnetic separation started as part of this project. In the first three years, Akai and Yamashita conducted basic tests for model samples using a conventional batch type high-gradient magnetic separator, and established the basics of a LAP recovery method using the separator.<sup>[4]</sup> A press release on this research was issued in May 2011. This was right in the middle of the rare earth crisis (the peak price was in October 2011), and we received numerous inquiries from the lamp manufacturers. While there were many voices pushing practical realization, the construction of a system contiguous to actual waste fluorescent powder processing became an immediate topic, and this led to the development of a device in the later NEDO project (2012-2013).

### 3 Topics in development of a separator

3.1 Mechanism of a high-gradient magnetic separator Responding to the request of the NEDO Project under which basic research had already started, the author (Oki) joined in the development of the device. First, the principle of a highgradient magnetic separator will be explained. The strength of magnetic trapping in the magnetic separator is generally expressed by the magnetic flux density (B) (unit, T: tesla; 1 T = 10,000 gauss) of the magnet surface. The surface magnetic flux density of a neodymium magnet is about 0.35 T, and about 1 T can be achieved partially when the magnetic circuit is established. Since electromagnets demagnetize at high temperature, it is normally about 2 T. On the other hand, a superconductive magnet can achieve magnetic flux density of over 10 T. Many of the magnetic separators are open systems, and the magnetic flux density declines inversely to the square of the distance. While it is not described as a specification, since the magnetic gradient (difference between magnetic flux density between two points) cannot be controlled, the "magnetic force" that actually attracts the particles is the product (B  $\cdot \Delta B$ ) of magnetic flux density (B) and a magnetic gradient ( $\Delta B$ ) (Fig. 1 top).

On the other hand, the attraction to magnets on the particle side is expressed as the magnetic susceptibility (dimensionless number) per unit volume. This is an index that expresses the property of a substance, but the attraction to magnets is also dependent on the particle volume, and the particle is more easily attracted to magnets as it increases in size. In the case of separating a mixture of various substances as in mines or recycling, it is extremely difficult to accurately separate fine particles of 50 µm or smaller according to properties (specific gravity, magnetic force, etc.) of the particle bulk. When sorting such fine particle mixtures, slurry that is particles dispersed in water is used to simplify the transportation and separation. However, water viscosity or the property of the particle surface becomes dominant in fine particles, and it becomes difficult to generate differences in particle motion according to particle bulk properties. However, in an earlier study by the authors (Akai and Yamashita), it was confirmed in the lab test that highly precise LAP recovery was possible using magnetic levitation by superconductive magnets, from the fluorescent powder mixture with particle diameter of about 5 µm that did not have strong magnetic force. This magnetic levitation is the same mechanism as the suspension magnetic separator, and is an open system. That means, with magnetic force equivalent to about 10 T in an open system, 5 µm of LAP can be selectively recovered. To construct a practical system at low cost, it is necessary to produce the same magnetic force at normal conductivity. In theory, if a magnetic gradient of about five times the open system is produced, it is possible to produce a magnetic force equivalent to 10 T in an open system, and the only method that can achieve this is a high-gradient magnetic separator.

Even if a magnetic body is placed in a position where 1 T parallel magnetic flux is produced when the N and S poles are set against each other, the particles do not move even in this extremely strong magnetic field. That is because there is no magnetic gradient (Fig. 1 bottom). Conversely, if a high magnetic gradient is produced, a strong magnetic force is produced even if the magnetic flux density is low. Although such an interpretation existed for a long time, H. H. Kolin et al. of the Massachusetts Institute of Technology proposed a mechanism for a high-gradient magnetic separator that actually realizes the phenomenon in 1968. This was the earliest case in which a separation mechanism itself was developed, and in recent years many physical separators have been developed. Kolin et al. succeeded in generating an extremely high magnetic gradient around thin wire by arranging the matrix of highly permeable thin wire in the parallel flux to concentrate the magnetic flux. This technology was realized as a de-ironing method (removal of colored minerals) of kaolin that is used in white pigment. In the 1980s, it was also applied to desulfurization/separation of coal (removal of sulfide iron

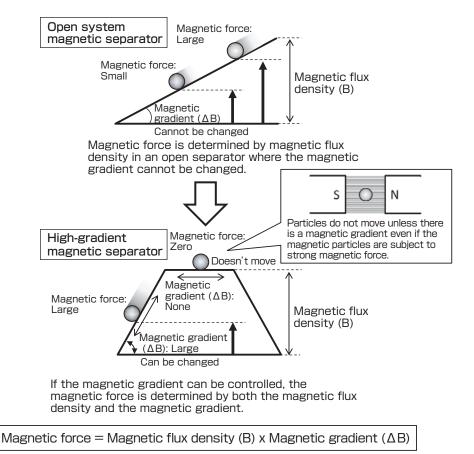


Fig. 1 Image of magnetic force in open system magnetic separator and highgradient magnetic separator

minerals). Until then, the floatation method using particle surface properties was mainly used for sorting of particles 50  $\mu$ m or less. With the development of this technology, the road to fine particle sorting was opened for sorting technology that utilized the particle bulk properties.

# 3.2 Topics of fluorescent powder sorting and development goals

Although a high-gradient magnetic separator has potential of sorting fluorescent powder at normal conductivity, there were two issues in developing the actual separator. The first issue is that this separator can provide a strong magnetic gradient but it cannot be precisely controlled. The expanded metal (the metal plates in which cuts are made and pulled to form diagonal nets) are used as a matrix, but the magnetic gradient is fixed according to magnetic flux density by magnetic permeability, thickness, special distribution between the thin wire, etc. of the material. That is, it is not possible to have control such as achieving desired magnetic flux density by voltage. Moreover, because there is unevenness of the magnetic force within the space and the matrix contracts due to excitation, it is impossible to accurately predict the unevenness. If the matrix is in a rough condition, the magnetic force the particle receives may differ depending on its travel route. Therefore, slurry is run in a dense matrix condition, all particles are attached to the matrix, and nonmagnetic particles are washed off in an excited condition. However, the engulfment of particles is not resolved and highly precise sorting is difficult. The second issue is the size of the device. Since the device was developed for use at mines, the continuously operating commercial separator is a large device, while the bench size device is a batch type lab

device. Considering the volume of fluorescent powder to be processed, a lab device is sufficient in terms of size, but it was necessary to newly develop a continuously operating device based on the lab device.

De-ironing of kaolin and desulfurization of coal are for the removal of impurities (magnetic recovery of substances to be removed), and the objective is to recover fine particles of iron (sulfide iron) as much as possible, by generating a strong magnetic force. There is no problem if some kaolin or coal was removed together with the impurities, and the objective is achieved even if the sorting selectivity is not that high. On the other hand, for fluorescent powder sorting, the idea is to selectively recover (magnetic recovery of valuable substances) LAP as a valuable substance. It was necessary to set a sorting limit between the slight difference of magnetic permeability of LAP and other particles, and the mission of the NEDO Project assigned to the author (Oki) was to achieve a prototype of a small continuously operating device in two years.

# 4 Automatic continuous operation of a small high-gradient magnetic separator

# 4.1 Outline of the automatic continuous operation system and comparison to the batch process

Based on the commercial batch type, a small high-gradient magnetic separator (a Jones wet strong magnetic separator, maximum 1 T), we aimed for the development of a magnetic separation system that could recover LAP at high precision, while allowing automatic continuous operation. Figure 2 shows the batch process and the newly developed automatic

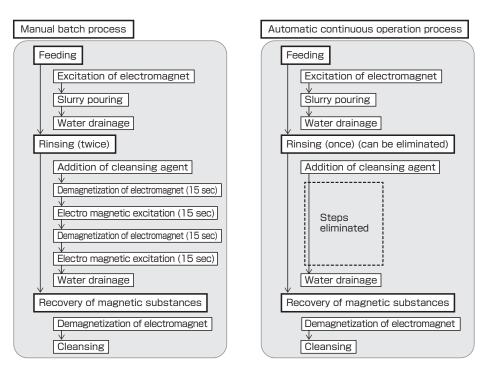


Fig. 2 Flow of conventional batch process and newly developed automatic continuous operation

	Recovery rate [Distribution ratio (%)]					Purity [Grade (%)]							
test		Magnetic substance	Rinsed substance	Nonmagnetic substance	Total		Before magnetic separation	Magnetic substance	Rinsed substance	Nonmagnetic substance	As pretreatment, the following three processes are conducted: (1) Sieving at 38 µm opening (recover undersized material)		
batch t	Overall (yield)	18.0	20.5	61.5	100						(2) Magnetic separation to remove iron (0.05 T magnetic separation,		
oa	LAP	86.4	4.8	8.7	100	LAP	12.0	60.7	3.0	1.8	nonmagnetic substance recovery)		
	BAM+CAT	23.6	17.9	58.5	100	BAM+CAT	4.0	5.1	3.4	3.7	(3) Sedimentation and cleansing (remove upper layer) three times		
in	SCA	4.9	18.2	76.9	100	SCA	6.5	1.9	6.2	8.7			
Manual	YOX	9.5	21.2	69.3	100	YOX	13.6	8.0	15.7	17.1			
2	Halo-phosphate	7.2	24.7	68.1	100	Halo-phosphate	60.7	23.4	70.9	64.9			
	Glass	5.5	6.2	88.4	100	Glass	3.2	0.8	0.8	3.8			
sno		Magnetic substance	Rinsed substance	Nonmagnetic substance	Total		Before magnetic separation	Magnetic substance	Rinsed substance	Nonmagnetic substance	As pretreatment, only one process is conducted: (1) Sieving at 20 µm opening (recover undersized material)		
ntinuous test	Overall (yield)						magnetic				is conducted:		
continuous on test		substance	substance	substance	Total	LAP	magnetic				is conducted: (1) Sieving at 20 μm opening (recover		
ic continuous ation test	(yield)	substance 17.9	substance 0.0	substance 82.1 20.2 72.3	Total 100 100 100	LAP BAM+CAT	magnetic separation	substance 62.9 9.7	substance 0.0 0.0	substance 3.5 5.5	is conducted: (1) Sieving at 20 μm opening (recover		
atic continuous eration test	(yield) LAP BAM+CAT SCA	substance 17.9 79.8 27.7 4.9	substance 0.0 0.0	substance 82.1 20.2	Total 100 100 100 100	BAM+CAT SCA	magnetic separation 13.9 7.3 4.7	substance 62.9 9.7 1.5	substance 0.0	substance 3.5 5.5 6.4	is conducted: (1) Sieving at 20 μm opening (recover		
imatic continuous operation test	(yield) LAP BAM+CAT SCA YOX	substance 17.9 79.8 27.7 4.9 7.0	substance 0.0 0.0 0.0 0.0 0.0	substance 82.1 20.2 72.3 95.1 93.0	Total 100 100 100 100 100	BAM+CAT SCA YOX	magnetic separation 13.9 7.3 4.7 16.9	substance 62.9 9.7 1.5 7.7	substance 0.0 0.0 0.0 0.0 0.0	substance 3.5 5.5 6.4 22.3	is conducted: (1) Sieving at 20 μm opening (recover		
itomatic continuous operation test	(yield) LAP BAM+CAT SCA YOX Halo-phosphate	substance 17.9 79.8 27.7 4.9 7.0 5.8	substance 0.0 0.0 0.0 0.0	substance 82.1 20.2 72.3 95.1 93.0 94.2	Total 100 100 100 100 100	BAM+CAT SCA YOX Halo-phosphate	magnetic separation 13.9 7.3 4.7	substance 62.9 9.7 1.5	substance 0.0 0.0 0.0	substance 3.5 5.5 6.4	is conducted: (1) Sieving at 20 μm opening (recover		
Automatic continuous operation test	(yield) LAP BAM+CAT SCA YOX Halo-phosphate Glass	substance 17.9 79.8 27.7 4.9 7.0 5.8 6.4	substance 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0	substance 82.1 20.2 72.3 95.1 93.0 94.2 93.6	Total 100 100 100 100 100 100 100	BAM+CAT SCA YOX Halo-phosphate Glass	magnetic separation 13.9 7.3 4.7 16.9 54.7 2.3	substance 62.9 9.7 1.5 7.7 17.0 0.7	substance 0.0 0.0 0.0 0.0 0.0 0.0 0.0	substance 3.5 5.5 6.4 22.3 60.0 2.2	is conducted: (1) Sieving at 20 μm opening (recover		
Automatic continuous operation test	(yield) LAP BAM+CAT SCA YOX Halo-phosphate	substance 17.9 79.8 27.7 4.9 7.0 5.8	substance 0.0 0.0 0.0 0.0 0.0 0.0	substance 82.1 20.2 72.3 95.1 93.0 94.2	Total 100 100 100 100 100	BAM+CAT SCA YOX Halo-phosphate	magnetic separation 13.9 7.3 4.7 16.9 54.7	substance 62.9 9.7 1.5 7.7 17.0	substance 0.0 0.0 0.0 0.0 0.0 0.0	substance 3.5 5.5 6.4 22.3 60.0	is conducted: (1) Sieving at 20 μm opening (recover		

### Table 1. Results of preliminary sorting test for batch device and automatic continuous device

Expanded metal is used as matrix for both tests.

\*LAP (LaPO4: Ce,Tb), BAM (BaMgAl10017: Eu<sup>2+</sup>), CAT (CeMgAl11019: Tb<sup>3+</sup>),

SCA ((SrBaCa)10(PO4)6Cl12: Eu), YOX (Y2O3: Eu3+)

continuous operation flow. There are several points that differ from the batch type, but the main difference is that in the batch type, the nonmagnetic substances are released in the water phase through repeated demagnetization and magnetization, while in the continuous system, the nonmagnetic substances are washed away with water only. Later, it was found that a rinsing effect appeared when the slurry flow rate was optimized, and sufficient sorting precision could be maintained even when the rinsing process was entirely eliminated. To verify this system, three sets of automatic, quantitative water supply and drainage systems and an automatic continuously operating high-gradient magnetic separator that incorporated those systems were fabricated (Fig. 3).<sup>[5]</sup> To optimize the system, first, preliminary investigation was conducted to see whether the continuous system would have the sorting precision equivalent to the batch system. In the batch system, three processes were

conducted as pretreatment: (1) sieving at 38 µm opening, (2) magnetic separation to remove iron, and (3) precipitation and cleansing three times. On the other hand, in the automatic continuous system, it was found that major simplification of pretreatment could be done by precise control of the supply flow rate and time and sieving at 20 µm opening. Table 1 shows the results of the batch system and the automatic continuous system using the expanded metal. Even when the processes of (2) and (3) were eliminated, in the automatic continuous system, the residues of iron, binding agent, and alumina powder were very small. Comparing the sorting results of the two systems, the recovery was 6.6 % lower in the continuous system while the LAP purity (grade quality) was 2.2 % higher in the continuous system, and it was confirmed that approximately the same sorting performance could be obtained as the batch system, even in the continuous system from which most of the pretreatment processes were

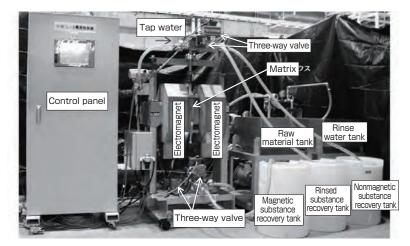


Fig. 3 Automatic continuously operated high-gradient magnetic separator (prototype)

eliminated.

# 4.2 Control optimization using a conventional matrix (expanded metal)

A commercial expanded metal matrix is shown in Fig. 4. The stacked expanded metal is inserted in the column so the slurry flow is interrupted somewhere within the matrix. The particles collide with the thin wire without exception, and the magnetic substances are captured. The nonmagnetic substances will attach to the matrix on the thin wire or become sandwiched between the magnetic substances, but are sorted after being washed by slurry or rinsing water. That is, whether the particles are recovered as "magnetic substances" after accumulating in the matrix or are recovered as "nonmagnetic substances" after falling off is determined by the relationship between the magnetic force and the flow rate of water. However, since it was initially developed as a device to remove magnetic impurities, there was not much consideration for the rinse flow rate control to improve the purity of magnetic substances, and this function was not incorporated in the commercial devices. In order to appropriately separate the particles with slightly different magnetic susceptibility into magnetic and nonmagnetic substances, it is necessary to control both the magnetic flux density and the fluid rate and time. Due to intellectual property concern, we shall omit the details of the investigative process, but after repeating various tests, we conducted a test to determine the optimal magnetic substance volume in one cycle. Figure 5 shows the magnetic substance volume of the matrix in one magnetic separation cycle, and the separation efficiency of LAP and non-LAP substances. The separation efficiency reaches maximum at around 50 g of magnetic substance volume. The magnetic force

distribution of the matrix is extremely uneven, and initially, non-LAP substances attach to the sites where the magnetic force is extremely strong, and the selectivity somewhat decreases. Later, when a certain amount is attached, magnetic attachment occurs at the site where the magnetic force is even, and the selectivity improves. However, as the magnetic substance volume increases, the magnetic force decreases as the magnetic and nonmagnetic substances are stacked onto the matrix, and the desired LAP substances are no longer magnetically attracted and the separation efficiency decreases again.

From the above results, the amount of magnetic substances attached to the expanded metal per cycle used in this test was determined to be about 50 g. The results of the systematic sorting test when the magnetic attached volume is set at 50 g are summarized in Fig. 6. In the first magnetic separation (rough sorting), the LAP purity increases from 16.5 % to 61.9 %. The LAP recovery is 79.4 %, followed by BAM + CAT 27.3 % and iron oxide 15.2 %. Other components are all 8 % or less, and at this point 92-95 % are removed as nonmagnetic substances. When the magnetic separation (fine sorting) is applied again to magnetic substances, the total LAP recovery decreases to 64.2 %, but the LAP purity increases to 82.0 %. When this is repeated again (fine resorting), the total LAP recovery decreases to 56.4 %, but the LAP purity increases to 85.9 %, and the removal rate of SCA, YOX, and halo-phosphate fluorescent powders reaches 99 % or higher. From the above results, it is determined that the appropriate condition when LAP recovery is prioritized should be "magnetic separation conducted once," and when LAP purity is prioritized, "magnetic separation conducted twice," considering the decrease in recovery. These were

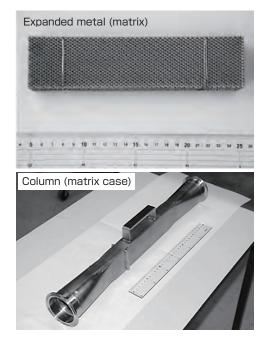


Fig. 4 Expanded metal (matrix) and column (matrix case)

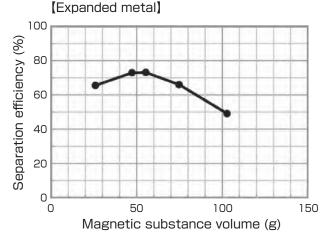


Fig. 5 Magnetic substance volume and selectivity of LAP (expanded metal used)

incorporated in the program of the automatic system.

### 5 Development of a high-selectivity matrix

### 5.1 Outline of a high-selectivity matrix

In the previous chapter, we developed an automatic continuous operation system using a traditional matrix to bring out its maximum performance, but we also developed a matrix with high selectivity to prepare for cases in which there is not enough sorting precision even when the developed system is used. In general usage of magnetic separation, the magnetic susceptibility difference between magnetic and nonmagnetic substances is extremely high, and in practice, it becomes the separation of magnetic and nonmagnetic particles (Fig. 7). Even in a high-gradient magnetic separator, the emphasis is on the removal of magnetic fine particles using strong magnetic force, and the unevenness of the magnetic force within the matrix is not considered. On the other hand, although LAP has the highest magnetic susceptibility among fluorescent powders, it is a weak magnetic body, and the difference in magnetic susceptibility from other fluorescent powders is small. Therefore, to improve the selectivity, we started to develop a matrix with a new way of thinking. In the matrix developed in this research,<sup>[6]</sup> the particles do not mechanically collide as in the conventional matrix, but even magnetic force is produced in the flow channel, and when the particles pass through the channel without barriers, only the particles with certain

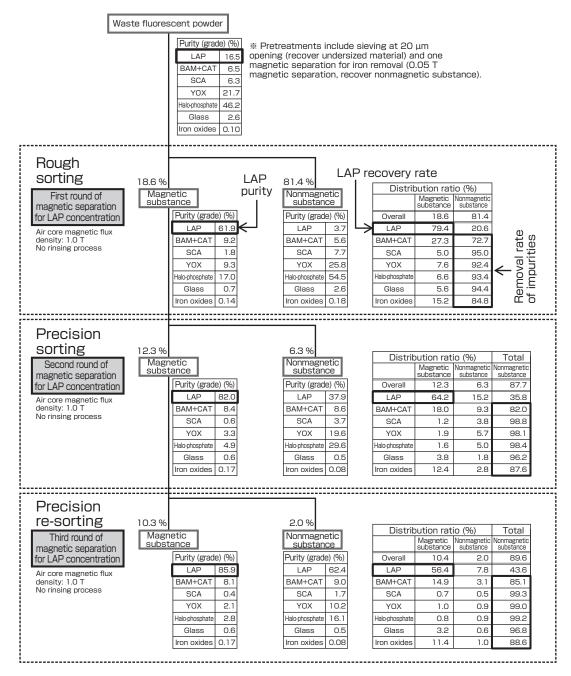


Fig. 6 Results of magnetic separation to obtain highly pure LAP using expanded metal

magnetic susceptibility are attracted and attach to the matrix by magnetic force. Therefore, the particles with magnetic susceptibility less than a certain threshold pass through the matrix, and it is expected that highly selective separation can be achieved. The developed matrix (Fig. 8) is designed by optimizing the internal magnetic force distribution through magnetic field simulation by a finite element method. It has a structure in which the waveform magnetic body walls of high rigidity and with 1 mm or lower height difference are placed facing each other, and regular and almost even magnetic force can be generated in the matrix space. It is also designed so the magnetic force strengthens only near the wall surface, to ensure the magnetically attached particles will not detach. As an example of the calculation results, the magnetic flux density (B) distribution around the matric under device magnetic flux density 0.9 T, and the magnetic force (B  $\cdot \Delta B$ ) distribution within the matrix are shown in Fig. 9. During the NEDO Project there was regular unevenness in the magnetic force distribution, but in the developed latest matrix, we succeeded in achieving almost even magnetic force, and now,

an extremely precise sorting threshold setting is possible.

### 5.2 Sorting performance of new high-selectivity matrix

To understand the selectivity property of the new matrix, we investigated the optimal magnetic substance volume of one cycle while setting the condition to be the same as the expanded metal of the previous chapter. The magnetic substance volume of the new matrix and the separation efficiency of LAP and non-LAP substances are shown in Fig. 10. Compared to the expanded metal, there is less contact opportunity with the particles, so the separation efficiency when the magnetic substance volume is low is a slightly low value. However, the separation efficiency hardly decreases even when the attachment volume reaches 50 g or more, and maintains 70 % even around 180 g. This is thought to be because constant long-range magnetic force occurs where the particles are attracted to the wall surface no matter where the particles are in the matrix space, and also LAP does not detach even when the magnetic substance forms a layer on the wall due to the strong magnetic force near the

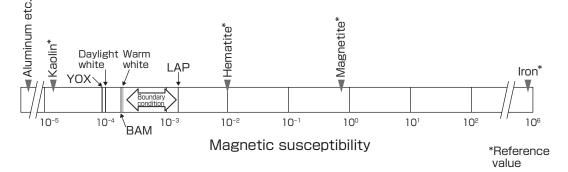
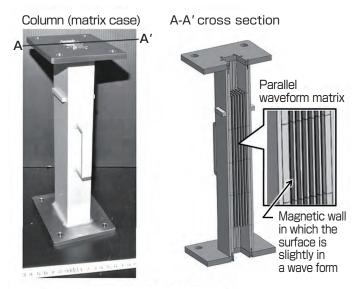


Fig. 7 Magnetic susceptibility of various substances and sorting boundary conditions for this research



Structure in which the wave plate runs parallel to the water flow

Fig. 8 Newly developed high-selectivity matrix

wall surface. Table 2 compares the selectivity results of the conventional expanded metal and the new matrix. From the results of the preliminary test, three cases of conditions prioritizing recovery and purity are given for both matrices. Although there are no great differences between the recovery and separation efficiency of the two, for the LAP purity and LAP concentration ratio, the new matrix shows about 10–30 % better results for both values.

On the other hand, there is a weakness, in principle, in this matrix. Conventionally, fluid transports the particles, but in the new matrix, the particle itself must reach the matrix by its own motion. Therefore, with finer particles with slow particle velocity, the time required for one cycle increases. While there is hardly any effect on particles of several ten µm or more that is the normal target of magnetic separation, waste fluorescent powder has extremely small particle size

of about 5 µm, and it is necessary to set the slurry flow rate to about 1/20 of the expanded metal. However, with the new matrix, there are two possibilities to supplement this. One is that the magnetic substance volume per cycle can be increased. As mentioned earlier, if the size is the same, about four times the amount of substances can be captured and recovered compared to the expanded metal. Second is that the generated magnetic flux can be distributed without loss in the matrix space, and the output of the electromagnet needed to capture LAP can be greatly reduced. As shown in Table 2, to obtain the same sorting results, the expanded metal requires 1.0 T (output 73 %), while the new matrix requires only 0.4 T (output 15 %). That is, if 1.0 T equivalent to the former is generated in the new matrix, a matrix with greater capacity can be utilized. The fluorescent powder processing volume per hour is 12.9 kg/h for the expanded metal, and 1.1 kg/h, or about 1/12, for the new matrix, and the same processing

after the NEDO Project

Air core magnetic flux density of electromagnet: 0.9 T Matrix wall (magnetic force distribution)

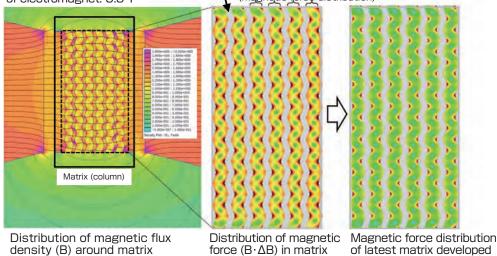


Fig. 9 An example of simulation result of magnetic flux density around high-selectivity matrix and magnetic force in the matrix

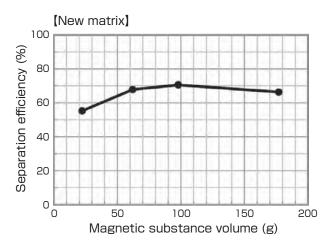


Fig. 10 Magnetic substance volume and selectivity of LAP (in new matrix)

	Expanded	metal						New ma	atrix				
condition	Air core ma flux density		1.0				condition	Air core magnetic flux density (T)		0.4			
	Feeding flo speed rate		1	1.2	1.45			Feeding flo speed rate			0.05		
Processing	Rinsing flor speed rate		No rinsing				Processing	Rinsing flow speed rate		なし	1.0	2.9	
Yield (%)			20.6	17.9	16.3		Yi	eld (%)	15.4	13.8	13.4		
LAP recovery rate (%)			84.2	79.8	78.8		LA	P recovery ra	ate (%)	83.1	79.7	73.0	
LAP separation efficiency (%)			73.1	72.0	72.1		LA	P separation effic	76.2	74.1	68.4	I	
LAP concentration ratio			4.1	4.5	4.8		LA	P concentration ratio		5.4	5.8	5.5	
Before separation			Substance after magnetic separation						Before separation		tance afte letic separ		
	LAP	13.9	53.0	62.8	64.2			LAP	13.9	60.2	64.5	70.1	
(%)	BAM+CAT	7.3	10.3	9.7	10.0		(%)	BAM+CAT	7.3	9.7	9.8	9.6	I
de)	SCA	4.7	1.8	1.5	1.4		de)	SCA	4.7	1.6	1.5	1.1	
(grade)	YOX	16.9	8.8	7.7	6.9		(grade)	YOX	16.9	6.9	6.5	5.2	
	Halo-phosphate	54.7	24.5	17.0	16.2			Halo-phosphate	54.7	19.5	15.9	12.3	
Purity	Glass	2.3	1.0	0.7	0.7		Purity	Glass	2.3	1.5	1.3	1.1	1
	Iron oxide	0.2	0.6	0.5	0.6			Iron oxide	0.2	0.6	0.6	0.6	
												$\Rightarrow$	-
		Priori <sup>.</sup> recov	ty on rery rate	Priority ( grade		n		Priori <sup>-</sup> recov	ty on Yery rate	Э	Prior grad		

Table 2. Results of high-purity magnetic separation for LAP using expanded metal and new matrix

LAP concentration ratio = LAP recovery rate/yield

volume can be obtained if the volume is increased 12 times.

The new matrix is still in development, but various characteristics and points that are superior to the conventional matrix have been demonstrated. Also, as mentioned earlier, development of higher precision type has been successfully created after the NEDO Project, and is being utilized in national projects for submarine hydrothermal deposits, copper ore de-arsination, and others. Compared to the conventional matrix, using the characteristic that allows sorting at very little magnetic susceptibility, it is expected to be used in various ways in the future.

# 6 Summary: Practical realization of the sorting system and the prospect for near-future resource circulation

In the years after the NEDO Project, automatic continuous operation of a small high-gradient magnetic separator was achieved by the author (Oki), and later, a license agreement was concluded with a magnetic separator company. During this time, the authors (Akai and Yamashita) conducted an investigation of the pre- and post-treatment processes. Also, a LAP lamp prototype test was done using waste fluorescent powder recovered as post-consumer products, and a result that luminous flux of 98.9 % could be achieved was obtained. There was no problem in the luminous flux maintenance rate, and it was found that LAP from which impurities were removed showed almost the same luminance as brand-new LAP, and was reusable. For this technology, Nomura Kohsan Co., Ltd., which is the major company that engages in waste fluorescent lamp processing and which also engages in mercury processing in Japan, was selected for the NEDO grant in 2014, and the developed technology was transferred to the Itomuka Mine of the company. From the original batch system to the development of the continuous system, the overview of the fluorescent powder sorting system that was installed in February 2015 is shown in Fig. 11. Flow from the beginning of this research to practical realization is summarized in Fig. 12. During this time, the "Minamata Convention on Mercury" was adopted in October 2013 and was concluded in February 2016, and there was great expectation for the Nomura Kohsan's ability to process mercury. On the other hand, in November 2015, the government announced a policy of promoting energy saving in lighting equipment, and the lighting equipment companies one after another announced termination of the production of fluorescent lamp lighting equipment. Although production of fluorescent lamp will be continued, it will be on a declining trend in Japan. The environment surrounding waste products is changing daily, and recycling technology must be able to adapt to the changes in society. Moreover, energy-saving properties of products should include possibilities for resource reuse and reduced energy consumed for disposal, as well as energy saving during manufacturing and usage. The age when a new product can be developed with an easy mind because recycling technology is established will arrive.

In this paper, the course of events was summarized from the

viewpoint of sorting device development, but some points should also be mentioned about how recycling is done. In general, there are two types of recycling. One is in-process recycling during manufacturing (of in-process waste), and the other is the post-consumer recycling (of post-consumer waste). In this research, both can be realized, but in general, the hurdle for the latter is extremely high compared to the former. As shown in Fig. 13, in-process recycling is the reuse of waste material that is process managed, and in most cases the purity is maintained to some degree. Although important as a production improvement process, these materials have never been used in society as products and do not contribute to resource circulation. On the other hand, the latter starts from a condition where waste products from various sources are mixed, and it is difficult to reuse the resource from this stage. The latter can be roughly divided into cascade recycling and horizontal recycling by methods of resource use. The waste disposal that Japan has been working on for a long time is a process where the objective is to not make products into waste material, and does not focus on their reuse. Although an extremely high recycling rate has been achieved, most of the recycling is cascade recycling.

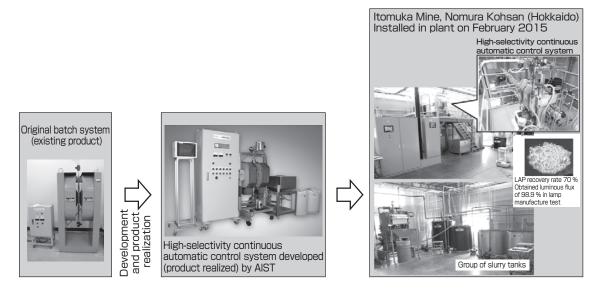


Fig. 11 Overview of the device from original batch system to fluorescent powder sorting system actually installed

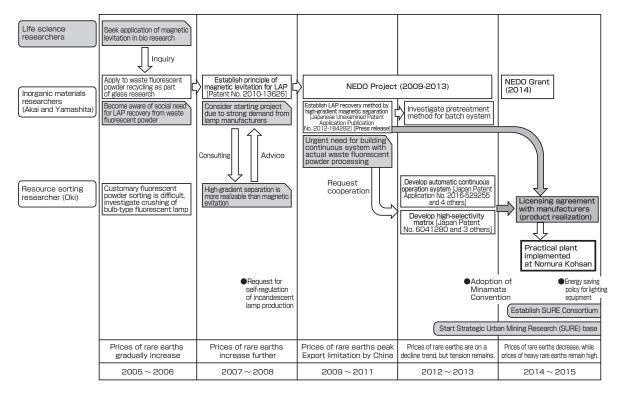
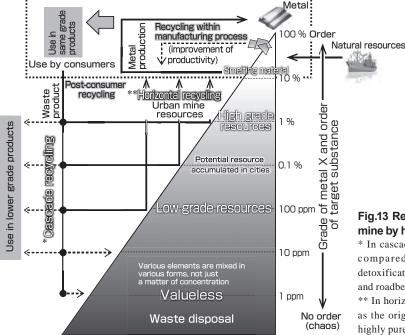


Fig. 12 Flow from beginning of research to practical realization

Particularly for metals, the main focus is on detoxification, and in many cases they are used as roadbed material and the values the metals possess is not really utilized. Even if cascade recycling increases, there is hardly any effect of reducing the import volume of natural metal resources, and in order to use waste products as urban mines, it is absolutely necessary to promote horizontal recycling. Fortunately, unlike organic materials, metal can be returned completely to original metal if it can be used as smelting material. However, metal excluding precious metals and copper does not have high value as raw material even if it undergoes troublesome recycling processes, and may not be feasible cost wise. The point that makes this research unique is that terbium metal is not recovered from the waste fluorescent powder but is made into LAP, raw material for high-functional materials (Fig. 14). A sorting process that enables reuse of waste fluorescent powder is the first in the world, but more than that, a case in which metal circulation is established not for metal but for high-functional material with higher value in the inner loop of horizontal recycling is almost nonexistent in the world. It leads as a system that can be considered an ideal circulation system for the near future by European CE/RE policies.

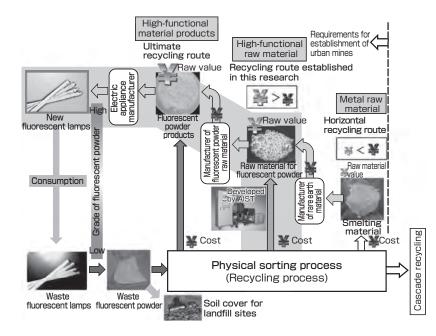
After many twists and turns, the advanced recycling technology for waste fluorescent powder described in this paper was brought to practical realization as several favorable conditions came about. On the other hand, for the establishment of future urban mines, it is necessary to



# Fig.13 Resource potential and establishment of urban mine by horizontal recycling

\* In cascade recycling, metal is reused in lower grade products compared to the original products. Its main purpose is detoxification, and the process includes mixing metal in cement and roadbed materials.

\*\* In horizontal recycling, resources are reused at the same grade as the original products. In case of metals, it involves reuse as highly pure metal through refining of smelting material.





place as many waste products on horizontal recycling. The cost is a major issue, but for cost reduction of recycling, the social system must change into one with a "circulating" loop in which products follow the artery-vein like cycle where products are made from raw materials and distributed (like the artery that carries oxygenated blood from the heart), and then products after use are collected as waste (like the vein which carries deoxygenated blood back to the heart), and reused and recycled like through the "artery." The authors established the Strategic Urban Mining Research (SURE) base at AIST in 2012, and created the SURE Consortium in 2013 with the collaboration of government and private companies. Currently, the members include 35 AIST researchers, 61 private companies, and 26 public research institutions, and they are engaging in activities for the construction of near future urban mines operated in a "circulating" loop. We hope the results of this research will provide hints as a leading system of near-future resource circulation allowing Japan to lead the world in urban mining development.

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### Authors

### Tatsuya OKI

Completed the doctor's course at the Graduate School of Science and Engineering, Waseda University in 1994. Doctor (Engineering). After working as Research Associate at Waseda University, joined the National Institute for Resources and Environment, Agency of Industrial Science and Technology in 1995. Principal



Research Manager, Environmental Management Research Institute, AIST since 2015. Engages in development of rare metal recycling technology such as tantalum condenser, and development of mineral ore processing for seafloor hydrothermal deposits. Participates in several NEDO and JOGMEC projects for resource processing technology. Established the Strategic Urban Mining Research (SURE) base at AIST in 2013, and started up the SURE Consortium in 2014. Currently, Chairman of the SURE Consortium. In this paper, was in charge of the development of high-gradient magnetic separator and matrix, as well as the overall structure of research.

### Tomoko AKAI

Completed the master's program at the Department of Inorganic Physics and Chemistry, Graduate School of Science, Osaka University in 1990. Withdrew from the doctor's program to join the Osaka National Research Institute, Agency of Industrial Science and Technology (currently AIST Kansai) in 1991. Received Doctor of Science degree in 1997 (Osaka



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### **Discussions with Reviewers**

#### 1 Overall

### Comment (Hiroki Yotsumoto, AIST)

This research succeeded in developing a recycling technology of rare earth fluorescent powder by applying a high-gradient magnetic separator used in the resource engineering field. I think it is significant that the researchers of different fields cooperated, set a clear goal, advanced the research in steps, and developed a new recycling technology.

### Comment (Chikao Kurimoto, AIST)

In modern society, recycling materials is one of the most important issues. This paper presents a success story on the advanced integration of knowledge through the collaboration of researchers specializing in materials and those specializing in sorting devices, toward the practical realization of recycling for rare earth fluorescent powder. The structure of the paper is clear, with presentation of detailed figures and tables. It appropriately describes the scenario from R&D to practical realization, and also discusses future prospect. I think the paper fulfills the objective of *Synthesiology*, and is appropriate for publication in this journal.

### 2 Scenario

#### Comment (Hiroki Yotsumoto)

I understand that it is important that the researchers of different fields cooperate, but you seem to emphasize the cooperation of different divisions inside AIST. This may not be necessary information for the readers.

I rather think it will be more valuable and interesting to the readers if you present how common knowledge and way of thinking of life science researchers clashed or blended with those of inorganic materials researchers and resource technology researchers. Can you provide us such stories?

### Answer (Tatsuya Oki)

I understood that this journal is different from usual journals where the results are presented matter-of-factly, and it asks for descriptions of researchers' roles, configuring of research, as well as the way of thinking and strategy that lead to a certain result. I revised the whole paper according to your comments.

On the other hand, pertaining to your latter indication, we did not seek optimal solution by integrating the opinions of all researchers. Rather, we passed on the issues that a researcher could not handle, in the order of life science  $\rightarrow$  inorganic materials  $\rightarrow$  resource technology, and the investigations for practical realization were fed back from resource technology  $\rightarrow$  inorganic materials. Therefore, there weren't any clashes. I think integration occurs when a problem is not left to an individual to solve, and the roles are played by the right people in the right places.

#### Comment (Chikao Kurimoto)

To show continuation from the research strategy released earlier (Oki, *Synthesiology* 2013), I think the argument will become clearer if you discuss by citing Oki (2013) in "Chapter 1 Introduction."

### Answer (Tatsuya Oki)

I added "At the time, the authors were conducting research and development of physical sorting of magnets and capacitors,<sup>[1]</sup> ...."

#### Comment (Chikao Kurimoto)

Figure 12 is important as it shows the flow of the scenario of this paper. In relation to the comment above, why don't you add the establishment of SURE as a movement that took place in Japan (or AIST)?

### Answer (Tatsuya Oki)

I added SURE and the SURE Consortium that are described in the text to the figure.

#### Comment (Hiroki Yotsumoto)

In the abstract, you use the expression, "there are few cases worldwide in which materials circulation from post-consumer waste has been established," but doesn't this contradict the fact that gold and platinum are recycled regularly? If you are saying the recycling described in this paper is of a different category from the recycling of precious metals, you must explain or else the readers will be confused.

### Answer (Tatsuya Oki)

The recycling of copper and precious metals falls into the category of "horizontal recycling" in which the recycled substance is reused at the same value as the original "metal" (as ingots). This research handles the "case in which metal circulation is established in a loop further inside horizontal recycling" (refer to Fig. 14). That is, waste fluorescent powder is not reused as "rare earth metal," but we made it usable as "fluorescent powder raw material" that has higher value. In the sense that it is returned to make products again, it seems to be "horizontal recycling," but normally, the highest concept of recycling is to return to the original metal form. This time, recycling was done to create a valuable material that surpasses that stage, and I think this is a rare case worldwide.

The point that you indicated was changed to "a case in which metal circulation is established not for metal, but for high-functional material with higher value in the inner loop of horizontal recycling."

# **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.

	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.

### Required items and peer review criteria (January 2008)

# 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, 1.1.1.2, 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:

Synthesiology Editorial Board

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 E-mail: synthesiology-ml@aist.go.jp

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

The copyright of the articles published in "Synthesiology" and "Synthesiology English edition" shall belong to the National Institute of Advanced Industrial Science and Technology(AIST).

Inquiries:

Synthesiology Editorial Board c/o Public Relations Information Office, Planning Headquarters, National Institute of Advanced Industrial Science and Technology(AIST) Tel: +81-29-862-6217 Fax: +81-29-862-6212 E-mail: synthesiology-ml@aist.go.jp

# Letter from the editor

The objective of Synthesiology is to accumulate specific case studies that illustrate which "scenario" is useful to utilize the results of research in society. A public testing and research institution established by a local government is generally called "kosetsushi," whose mission is to help the creation of innovation by small and medium-sized enterprises, and they stand at the forefront of the activities to return the research results to the local community. This issue carries the paper "Development of low sticky texture natto 'MAMENOKA"" that describes some of the activities of such a kosetsushi. Recently, natto has been gathering interest as a health food, but its consumption has leveled off. A clear goal of overseas diffusion was set, and a kosetsushi developed the technology to enable this. Moreover, for the project, a design researcher of a university and a chef of a famous restaurant were engaged, and by rallying their wisdom, a strategy for the producing region was created. This is a case study that further broadens the width of the "scenario" that Synthesiology has accumulated over the years. Starting with this paper, I hope there will be more papers submitted by the kosetsushi in the future.

It can be seen from other papers published in this issue that "local" and "fusion of different fields" are keywords of innovation creation. In "Submarine earthquake- and tsunamiinduced event deposits," the research to clarify the history of tsunami that can cause serious damage to the local community is being done through superb fusion of marine geological surveys, physical insight, and paleontological findings. The "High-accuracy endoscopic microscopy using a thin, 1.5 mm diameter probe with optical coherence tomography" describes the course of events where the passion for attaining top-ofthe-world performance by a precision machine company in Tohoku moved the hearts of optical technology researchers at AIST, and the unequalled core technologies of the two were fused to produce an innovative internal measurement technology. In "Materials recycling technology for recovering rare earth fluorescent powder from fluorescent lamp sludge," the magnetic levitation technology that was being studied by life science researchers was shown to be effective, in principle, as a method for sorting fluorescent powder from waste fluorescent tubes, and the researcher specializing in resource sorting technology established its industrial application. It is an excellent example where the technology was handed over organically from one field to the other, to produce a result.

Innovation is said to be new bonds and new departure for things. There is no question that the technological invention is important in industry, but it is also true that invention alone does not necessarily lead to business differentiation. The four papers in this issue well illustrate that a highly specialized researcher should not stop at simply executing R&D within his/her specialty, but it is important to generate innovation through new bonds with other specialists.

(Keiichi IKEGAMI, 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

### **Research papers**

Development of low sticky texture natto "MAMENOKA" Y. KUBO and R. NAKAGAWA

Submarine earthquake- and tsunami-induced event deposits

—Disturbance of the sea floor by huge earthquakes and their related tsunamis, and the use of disturbance records in marine sediments for the history of past huge earthquakes and tsunamis— K. IKEHARA and K. USAMI

High-accuracy endoscopic microscopy using a thin, 1.5 mm diameter probe with optical coherence tomography H. FURUKAWA, N. NOGUCHI, H. YAMAZAKI and T. ASADA

Materials recycling technology for recovering rare earth fluorescent powder from fluorescent lamp sludge *—Pioneering near-future resource circulation—* 

T. OKI, T. AKAI and M. YAMASHITA

Editorial policy Instructions for authors Letter from the editor Aim of *Synthesiology* 

"Synthesiology-English edition" is a translated version of "Synthesiology," which is published quarterly, ISSN 1882-6229, by AIST. Papers or articles published in "Synthesiology-English edition" appear approximately four months after the publication of the original "Synthesiology."