

# Synthesiology

English edition

**Development of a real-time all-in-focus microscope**

**Portable national length standards designed and constructed using commercially available parts**

**How the reliable environmental noise measurement is ensured**

**Bioethanol production from woods with the aid of nanotechnology**

*Synthesiology* editorial board

## MESSAGES FROM THE EDITORIAL BOARD

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

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

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

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

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

*Addendum to Synthesiology-English edition,*

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*Synthesiology* Editorial Board

**Note 1 :** The period was named “nightmare stage” by Hiroyuki Yoshikawa, President of AIST, and historical scientist Joseph Hatvany. The “valley of death” was by Vernon Ehlers in 1998 when he was Vice Chairman of US Congress, Science and Technology Committee. Lewis Branscomb, Professor emeritus of Harvard University, called this gap as “Darwinian sea” where natural selection takes place.

**Note 2 :** *Type 2 Basic Research*

This is a research type where various known and new knowledge is combined and integrated in order to achieve the specific goal that has social value. It also includes research activities that develop common theories or principles in technology integration.

**Note 3 :** *Full Research*

This is a research type where the theme is placed within the scenario toward the future society, and where framework is developed in which researchers from wide range of research fields can participate in studying actual issues. This research is done continuously and concurrently from *Type 1 Basic Research* (Note 4) to *Product Realization Research* (Note 5), centered by *Type 2 Basic Research* (Note 2).

**Note 4 :** *Type 1 Basic Research*

This is an analytical research type where unknown phenomena are analyzed, by observation, experimentation, and theoretical calculation, to establish universal principles and theories.

**Note 5 :** *Product Realization Research*

This is a research where the results and knowledge from *Type 1 Basic Research* and *Type 2 Basic Research* are applied to embody use of a new technology in the society.

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# Development of a real-time all-in-focus microscope

— WYSIWYG in the micro-world —

Kohtaro Ohba

[Translation from *Synthesiology*, Vol.2, No.4, p.264-275 (2009)]

In this paper, our struggle to realize a high-speed digital processed microscopic observational system for tele-micro-operation with a dynamic focusing system and a high-speed digital-processing system using the “depth from focus” criteria is reported. To realize the system, each functional element and its system configuration had been deeply discussed not only in the academic society but also with several companies, there were many trials and errors, and the final product system had been developed after several trials.

**Keywords :** Real-time, microscope, all-in-focus

## 1 Technological issue to be realized

Recently, there have been increase in industrial enthusiasm in micro- and nano-technologies, and there is also an increase in the demands for systems that can observe the microenvironment and systems that allow manipulators to operate the microenvironment. The examples of observation systems are optical and electron microscopes. The optical microscopes are widely used to observe micro-size objects that do not surpass the optical limit; i.e. sub micro meter, and lack of the depth information. In biological usage, the products demanded in the micro-size market include ones that allow cell and DNA manipulation while looking at the optical microscope images. In industrial usage, there are demands for observing and inspecting both the wire-bonded chip surface and bonding surface at the same time in LSI product inspection.

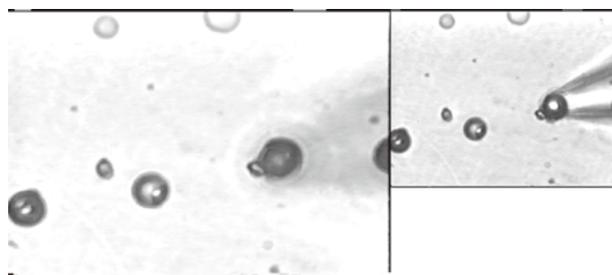
In such microscopic operation, it is necessary to conduct manipulations while sensing the three dimensional position of the actual micro-object. The major differences between an ordinary environment and a microenvironment are, in the latter, (1) the viscosity due to the van der Waals force rather than the weight of the object due to physical scaling effect cannot be neglected, and at the same time (2) as we approach the optical limit of the optical microscope, the range in the depth of the visible object (depth of field) becomes extremely small due to optical scaling effect. In this research, the issue of (2) optical scaling effect is addressed.

As a result of the optical scaling effect, in an optical system with shallow depth of field as in a microscope image, the objects with different depths are unfocused when an object with certain depth is set in focus, as shown in Fig. 1. Therefore, for product inspection in many cases, several images are shot at varying focal distances while moving the focal distance, and then these images are processed. Therefore, we set our objective as virtually reducing the issue of optical scaling

effect, and we set our technological issue as the construction of a “real-time all-in-focus microscope,” which is a new micro-visual system that solves the optical scaling effect within “real time” during microscopic manipulations in high-power optical systems such as the optical microscope<sup>[1]-[4]</sup>.

## 2 History of struggle: twists and turns

Here, we chronologically present the path from an idea that initially started as a scientific interest to product realization, while overcoming several feasibility study (FS) phases. The primary innovative idea was published in regular academic papers and as product articles after it was finalized as a product. For the *Synthesiology* paper, I shall explain the process from the idea phase through several FS phases and then finally to product realization. I shall specifically mention that the course of these FS phases was greatly affected by the “strategic deepening and selective synthesis” that are described as synthesiology including selections and rejections of some components, development of algorithms dedicated to those components, and meeting by chance with partners to realize them, as well as the “lucky coincidences” that couldn’t be quite categorized. That is because as we blindly wandered in the dark and finally arrived at the goal,



**Fig. 1 Microscope image of microscopic operation (manipulating 4  $\mu\text{m}$  glass beads).**

(a) Glass beads and forceps are at different heights, and (b) glass beads and forceps are at same height.

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only in retrospect can we say certain places were the forks in the road. While I was groping desperately, honestly speaking, I cannot say I did any strategic decision-making even in retrospect. Even if a theoretical order was given for the twists and turns, the situation differed greatly in the next blind wandering, and there were very few instances when I felt the previous experiences helped at all. Therefore, our history of twists and turns (Fig. 2) will be presented as an article that documents a process of product realization, not in the format of an academic thesis, and I shall explain the phases from a technological standpoint in the following sections.

**(a) Idea phase**

When a person looks at an object with the naked eye, the objects both close and afar seem to be in focus. This is accomplished by the focal adjustment of the eyes. In contrast, when one sees things through the lens such as of a camera, it is necessary to bring the lens into focus. The automatic focus camera sets the focus automatically, but it can do so at certain distance only.

In a microscopic environment, the depth of field can be increased by narrowing the aperture in the low-power single-lens reflex cameras and stereomicroscopes, but it is impossible to cover all areas as magnification increases due to the optical principle. In microscopic objects, the depth

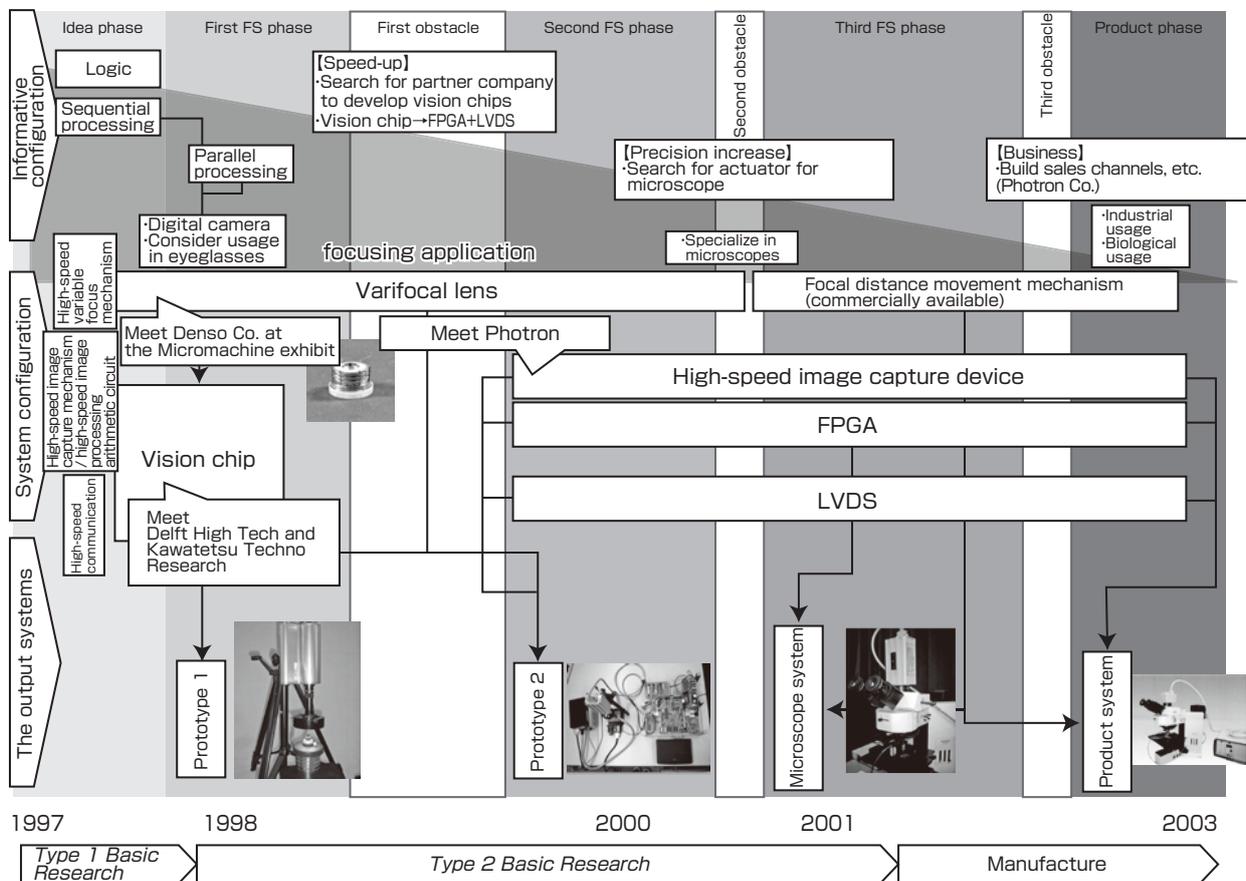
of the objects cannot be discerned through images with deep depth of field. Therefore, we thought of increasing the operability in the microenvironment by making use of the shallow depth of field.

To increase the operability in the microenvironment, we decided to create a system that fulfills the following two conditions simultaneously, and conducted theoretical considerations and devised processing algorithms:

1. Dynamic observation (30 frames/sec.) of real image with depth of field ideally raised to infinity
2. Real time measurement (30 frames/sec.) of three-dimensional shape of the object

At this time, we were vaguely thinking that it may be interesting to have this function in the digital camera or eyeglasses, and had no idea about any specific device application.

The above conditions 1) and 2) were not stated as initial target specifications, but as we were looking through papers for methods to obtain the image of 1) using the image processing technology, we realized that the 3D position information of objects were not used in the course of processing. Therefore, we thought why not use this information? The details will be discussed later.



**Fig. 2 Our struggle (Twists and turns) for synthesis.**

**(b) First FS phase**

To incorporate the idea into an actual hardware system, high-speed variable focus mechanism and information processing and communication technology to accomplish the data acquisition, communication, and processing in real time were required to synthesize the system. For the high-speed variable focus mechanism that allowed dynamic observation at 30 frames/sec., response rate at 30 Hz was required, but available products only had single-digit response. However, this was solved when we visited a micromachine exhibition and came across the varifocal lens developed by the Denso Corporation in their micromachine project. For information processing and communication, when the number of necessary images to composite a single image was  $N$ ,  $N \times 30$  frames/sec.  $\times$  image digital size would be necessary. We then met Delft High Tech Corporation (current DHT Corporation) that imported the vision chip MAPP series from the Netherlands, and Kawatetsu Techno Research Corporation (current JFE Techno Research Corporation) that was working on its development. We were able to solve both technological issues and created Prototype 1.

**(c) Second FS phase**

However, speed remained at 0.5 frames per sec., and the initial goal of 30 frames/sec. could not be achieved. We spent about a year negotiating with several companies to develop the high-speed vision chip. This was our first obstacle. In ordinary research, we could have written, "The problem can be solved by developing an ultra high-speed vision chip and implementing it to the device," and then leave it. Yet, we set out on the voyage to clear the first obstacle from the desire to carry this project out to the end. However, even if we visited the companies with our request, they turned us down because they did not see any business merits for spending several hundred million yen for the development. The break came when we visited Photron Ltd. This was a high-speed camera manufacturer that had technology to shoot images at 10,000 frames/sec. and to transfer them to memory. They gave us encouraging advice that our requirement could be realized by a high-speed image capture device and the LVDS (low voltage differential signaling)<sup>Term 1</sup> interface and by using the FPGA (field programmable gate array)<sup>Term 2</sup> without developing the vision chip. Prototype 2 was created in a month.

**(d) Third FS phase**

In this phase, we started discussing future business with Photron. When we participated in exhibitions, we learned that the all-in-focus image was most highly demanded in microscope use, and therefore we started developments specializing in microscope application. Initially, the Denso varifocal lens was hand-made, mass production was not possible, and it was not suitable for high-precision positioning required for the microscope. We sought out the piezo-actuator of Physik Instrumente (PI) GmbH of Germany. In this device, the focal distance of the microscope was adjusted

electrically, and we could obtain the desired precision by modifying the control unit. The second obstacle was to achieve high precision in high-speed mechanical movement, and the third FS phase was to overcome this issue. Initially, we were thinking only about the high-speed information processing, and expected the high-speed mechanical movement would somehow fall into place. In fact, we were plagued to the end on how to achieve highly reliable high-speed mechanical movement.

**(e) Product realization phase**

By repeating several demonstrations using the microscope system, we considered further business application by separating the uses into industrial and biological. However, the microscope system had a special sales channel where the optical manufacturer visited the researchers to sell it and then became in charge of maintenance. For an outsider like Photron to enter, it had to provide OEM to conventional optical companies and establish the sales channel to reach the users. This was our final obstacle. Over one year was needed to establish the sales channel, and the product was launched into the world in 2003.

**3 Idea phase**

The idea explained in the previous section was born from a simple scientific interest of "wanting to make an image with everything in focus." The initial application of this function was vaguely considered for digital cameras and eyeglasses. We started by checking the efficacy by conducting offline processing on a PC for several images with different focal distances.

In this phase, to conduct simple processing within the memory capacity and processing ability of the PC, we developed a sequential processing algorithm to increase the memory efficiency and a parallel processing algorithm to increase the processing capacity.

**3.1 Theoretical synthesis - depth from focus method**

It was described earlier that in case of optical microscope image, the issue of shallow depth of focus greatly affected the operability. At the same time, this may be a major advantage to realize the depth from the focus method<sup>[6][7]</sup> which is one of the 3D measurement methods of objects<sup>[5]</sup>. While the all-in-focus camera was a solution to the shallow depth of focus, by making use of this problem, not only could we achieve an all-in-focus image where every part was in focus, but also by using the depth from the focus method, it became possible to obtain the 3D shape of the object using only a single lens.

Figure 3 shows the conceptual diagram of the depth from the focus method. When observing an object with different depths, the distance to the image surface is scanned, and the local frequencies of the dark-light data at each point of the

image are measured to detect the peak. Once the distance to the image surface in focus is obtained, the distance to the object can be calculated by using the Gauss' lens law.

Whether the focus is correct can be determined by conducting the local spatial frequency analysis around the observation point of the image while moving the focal distance  $f$ , object distance  $l$ , or image distance  $l'$ . The point with greatest spatial frequency is the point in focus. This method is used often in the automatic focus mechanism, and one can intuitively see that the out-of-focus area has low frequency while the in-focus area has high frequency. Basically, the focus of the lens is moved using the variable focus mechanism. The images are captured one by one, the local spatial frequency analysis around the pixel point is conducted for each image, the peak of the frequency or the area in focus are picked up from the images for each pixel, and these are pasted together as one image to obtain an all-in-focus image. The 3D data can also be obtained from the focal distance and image distance at each point.

There are many methods of assessing the degree of focus of an image such as looking at the changes in brightness of the image while changing the focal distance. In this paper, considering the final product realization, we define the following equation for Image Quality Measure (IQM), to assess the local spatial frequency analysis of each pixel through spatial dispersion of image brightness value, for the reason that the image-processing algorithm can be easily implemented in the hardware. This IQM value was originally defined as one of the indices that indicate the clarity of the image, and was not for determining whether the image is in focus or not. However, we decided to use the IQM value since the processing algorithm can be easily adapted to higher speed in the future, with the assumption that the image is digitized and will be digitally processed.

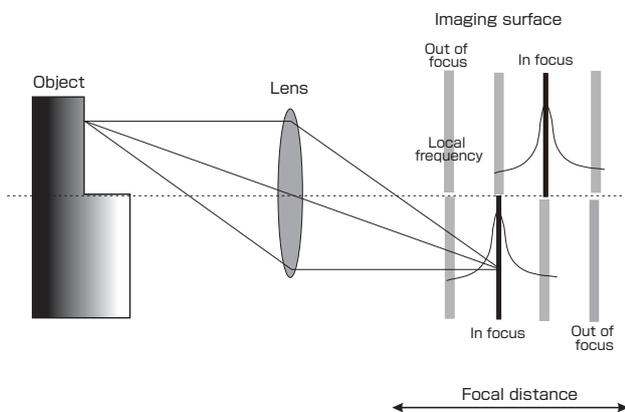


Fig. 3 Depth from focus method.

$$IQM = \frac{1}{D} \sum_{x=x_i}^{x_f} \sum_{y=y_i}^{y_f} \left\{ \sum_{p=-L_c}^{L_c} \sum_{q=-L_r}^{L_r} \left| I(x, y) - I(x+p, y+q) \right| \right\}$$

Here,  $(-L_c, -L_r) - (L_c, L_r)$  and  $(x_i, y_i) - (x_f, y_f)$  are small regions for conducting dispersion assessment and smoothing.  $D$  is the number of all pixels to which assessment is conducted for normalization at pixel unit. The IQM values are assessed for each pixel or region while moving the focal distance, the peak of the IQM value is detected, the object distance  $l$  is calculated from the focal distance  $f$  and image distance  $l'$ , and then this is substituted in the matrix component for each pixel position to create the 3D data of the object.

### 3.2 Configuration of sequential processing – for reduced load on memory

As mentioned before, it is theoretically possible to obtain both the all-in-focus image and the depth image simultaneously, using the depth from focus method. However, in 2000 when we started this development, when calculating the algorithm for the IQM value, 2 Mbyte image memory and capture and processing of 30 shots/sec.  $\times$  30 frames = 900 images (about 3 min. using the PC in 2000) were necessary to obtain one all-in-focus image and depth image from about 30 images of 256  $\times$  256 pixels in real time.

In case it is necessary to obtain  $N$  number of different depth images to capture an all-in-focus image and a depth image at 30 frames/sec., an image capture device with high dynamic range that can shoot at 30  $\times$   $N$  frames/sec. is required. Moreover, a high-speed processing system to process and display such volume of image data is required.

In an automatic focus camera, to obtain the IQM value, the values for one or few points can be calculated and the focus can be moved according to the value. However, to obtain the all-in-focus image, calculations must be done efficiently within 33 ms for each pixel point.

The method the authors devised for optimizing the memory constitutively through algorithm and for overcoming the limitation of this hardware will be described below. In the following chapter, the configuration for optimizing the processing speed using the hardware characteristics will be explained.

When conducting these IQM processing at all pixel points, it was not efficient to process by temporarily storing the different pre-images with varying focal distances. Therefore we constructed a configuration using the sequential algorithm with steps (1)-(7) as shown below. Figures 4 and 5 show the main system configuration diagram and the flow chart, respectively.

Using this sequential algorithm, sequential processing is conducted while changing the focal distances, and by moving the focal distance to the end, each of the matrixes of the finally updated image memory becomes the all-in-focus image (AIF) and the depth image (DEPTH). While the equation for IQM seems complicated, only Laplacian and smoothing processes are applied as image processing technology. Laplacian is a secondary differentiation, and in the world of digital image, differentiation is the difference from the neighboring pixel, and it becomes Laplacian by differentiating twice. Smoothing is averaging. The two processes are four arithmetic operations, and are optimal for hard logic circuit for high speed. Normally, the memory will hold N frames of differing depth images, the IQM image for each are calculated, and the all-in-focus and depth images are obtained (image memory for total  $2N + 2$  frames were necessary) by comparing the IQMs at the same pixel position. In contrast, in the sequential algorithm, the obtained images are sequentially compared on the spot, and the memory is needed only for four images consisting of the original image, IQM image, all-in-focus image, and depth image. This reduced the memory requirement, and it became easy to configure the hardware, as it will be explained later.

- (1) Format  
init (IQM);
- (2) Move focal distance  
for (FV=0 to FVMAX) { (FV: focal distance)  
mov (FV); move focal distance
- (3) Image input  
IO = input; image input
- (4) Laplacian filter  
IL = lap (IO); preliminary processing
- (5) Median filter (average filter)  
IM = ave (IL); preliminary processing
- (6) Comparison of brightness value of each pixel, and corresponding copy of image data  
if (IM(x, y) > IQM(x, y)) {  
    IQM(x, y) = IM(x, y); update assessment value  
    AIF(x, y) = IO(x, y); create all-in-focus image  
    DEPTH(x, y) = FV; create focal distance information  
}
- (7) Image data output  
output (AIF, DEPTH); image data output

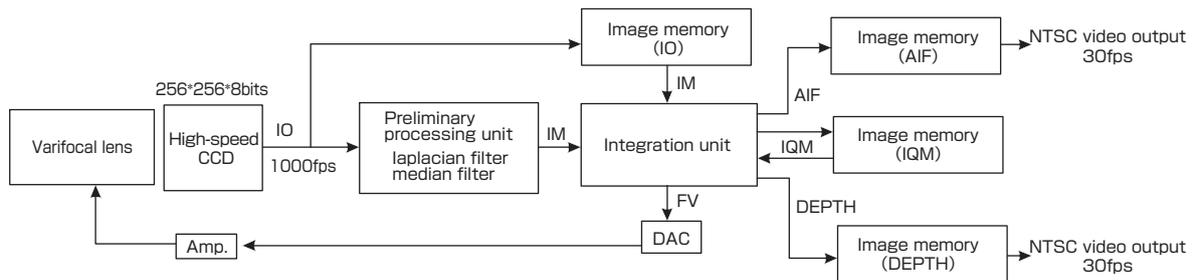


Fig. 4 System configuration for sequential algorithm.

#### 4 First FS phase

To accomplish the above methods and information processing in real time, it is necessary to move the depth of focus at high speed and at the same time, capture and process the images at high speed. Therefore, we determined that the basic system components were the following three:

- (a) high-speed variable focus mechanism
- (b) high-speed image capture mechanism, high-speed image processing arithmetic circuit
- (c) high-speed communication.

For example, in conducting the process in eight-step focal distance, to obtain the output in real time at 30 frames/

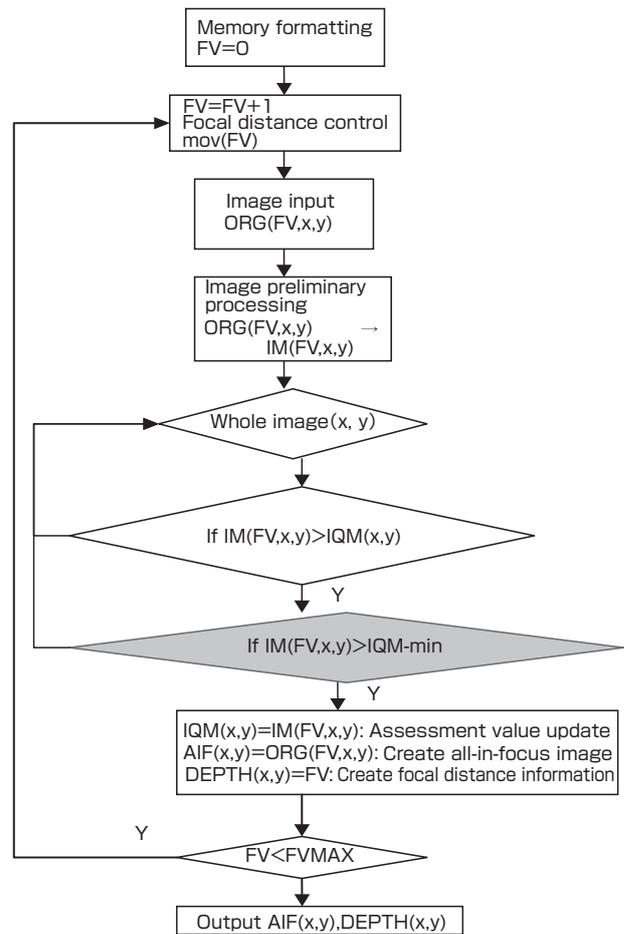


Fig. 5 Flowchart for sequential algorithm.

sec. that is sufficiently smooth for human observation, synchronized high-speed movement of the focal distance at 30 Hz and the image capture and processing speed of 240 frames/sec. at  $30 \times 8$  are required (Fig. 6). In case of 240 frames of black-and-white  $512 \times 512$  pixels, the pixel rate will be close to 100 MHz. At the same time, the focal distance of camera, the object distance, or the image distance must be moved at 30 Hz.

**(a) High-speed variable focus mechanism**

For Prototype 1 and Prototype 2, the varifocal lens developed by Denso Corporation was used as the high-speed variable focus mechanism<sup>[8]</sup>. It is driven by a piezo element, and the focal distance changes according to the voltage applied. The structure is simple without any motor. The focal distance is changed by moving the glass diaphragm using the bimorph actuator. By changing the voltage applied to the PZT bimorph, the lens can be changed from a convex to a concave lens. It has been demonstrated that the frequency response is possible up to around 150 Hz without delay in phase. Figure 7 shows the varifocal lens, Fig. 8 shows the structure, and Fig. 9 shows the details of the lens driving mechanism. When no voltage is applied, the lens is a planar glass. The greatest characteristic of this varifocal lens is its high speed. Since the glass diaphragm is directly driven using the piezo element, high-speed movement of the focal distance is possible.

**(b) High-speed image capture mechanism, high-speed image processing arithmetic circuit**

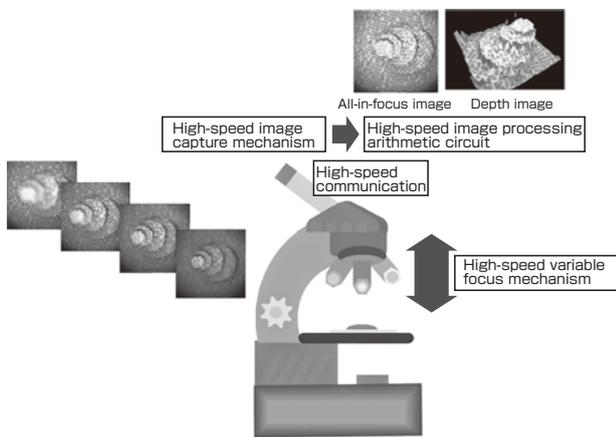


Fig. 6 System configuration for all-in-focus microscope.



Fig. 7 External appearance of varifocal lens.

For the high-speed image capture mechanism and the high-speed image processing arithmetic circuit in Prototype 1 of section 3.1, we used the vision chip that included an image capture device, ADC, and a processing system. By using this vision chip, processing was concluded within the vision chip, so high-speed communication was unnecessary. Here, since the volume of image data per unit time and image processing capacity were high, we used the column parallel type vision chip, MAPP2200, from Integrated Vision Products AB of Sweden. The basic configuration was CMOS image sensor with  $256 \times 256$  pixels, 256 ADCs, and 256 parallel processors. As mentioned in the previous chapter, using the image-processing algorithm that allows SIMD (single instruction multiple data)<sup>Term 3</sup> processing, the image capture and processing can be done at high speed.

**(c) Configuration with parallel processing**

In 2000, when we started the development, although the image-processing technology at an ordinary frame rate was already in practical use, the image capture and processing at a frame rate one digit higher would not be realized unless a special vision chip was developed. Of course, we were told that development of such vision chip required about a hundred million yen in cost. We visited several companies, but none gave us encouragement. In Prototype 1, we used the general-use vision chip MAPP2200 that was developed in Sweden to implement the parallel processing algorithm. Two-second processing speed was realized, but this was far from the real-time processing at our target frame rate.

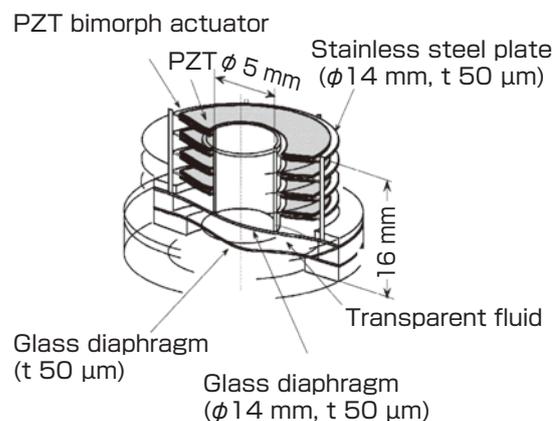


Fig. 8 Configuration of varifocal lens.

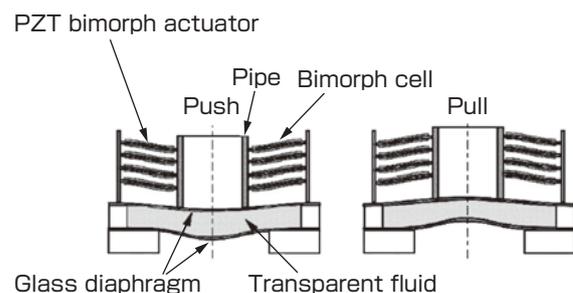


Fig. 9 Principle of varifocal lens function.

High processing capacity was required for the Laplacian filter and smoothing filters in the preliminary processing of the image in this method. Figures 10 and 11 show the hardware version of this process, which is an example of the vision chip implemented in Prototype 1 that will be explained later in this chapter. For the hardware used, the chip allows easy SIMD processing, and therefore, to calculate the above IQM value, as a process using the pixel values around each pixel points, parallel calculation of the Laplacian and smoothing filters are necessary. The examples of SIMD processing for each operation are shown. Looking at each calculation result, Laplacian filter (R12 of Fig. 10) and smoothing (R5 of Fig. 11) were ultimately obtained for each pixel point.

For the final product realization, while using this processing method as a reference, we configured a system that calculated the IQM value using the FPGA. The external appearance of Prototype 1 system created in 1999 is shown in Fig. 12.

As an assessment test, we conducted processing by placing an object with a depth of 35 mm at a position of 160 mm from the varifocal lens. To move the focal distance to cover an object with a height of 35 mm, 21 images were captured, and the resolution of depth was 1.67 mm. While the spatial resolution of the system is dependent on the setting of the optical device, in this case, since 16 mm × 16 mm was processed at an image resolution of 256 × 256, the resolution was 62.5 μm/pixel.

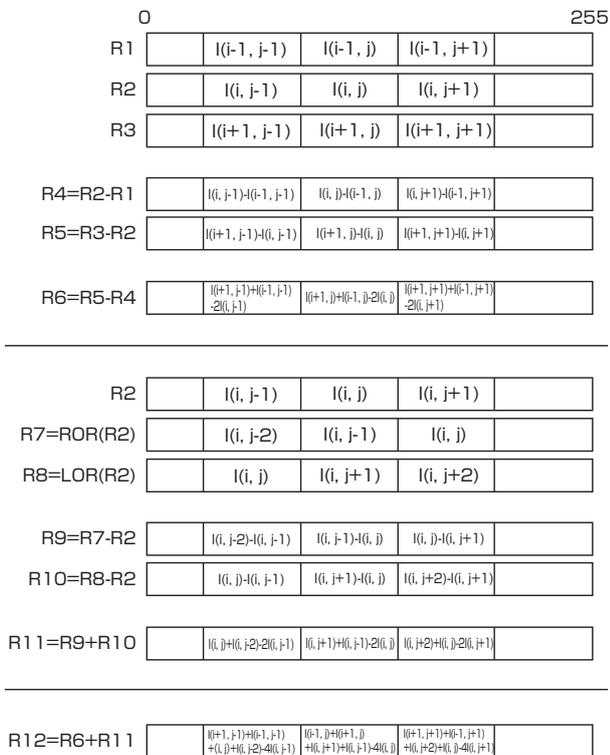


Fig. 10 Laplacian filter processing operation by SIMD processing.

The object used here was an artificial four-step pyramid of a height of 35 mm. The first step was φ10 mm and a height of 10mm, the second step was φ7 mm and a height of 10 mm, the third step was φ4 mm and a height of 10 mm, and the fourth step was φ3 mm and a height of 5 mm.

Part of the 21 images shot while moving the focal distance is shown in Fig. 13 on the left. As mentioned earlier, by conducting processing, the all-in-focus image as in Fig. 13 on the right and the VR display as in Fig. 14 were obtained. While the all-in-focus image itself was adequate, we obtained a largely dispersed result since the depth image had a small region that required smoothing, and the resolution in the depth direction was small.

While the performance of Prototype 1 was dependent on the

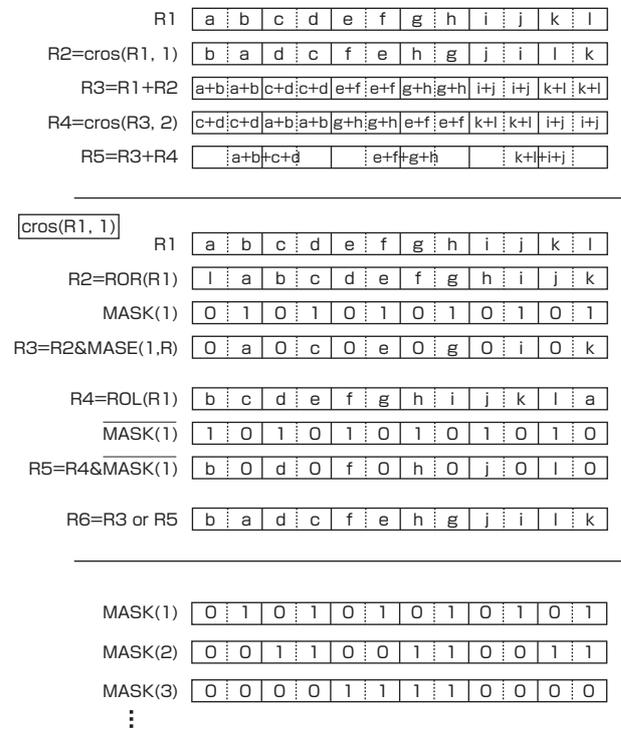


Fig. 11 Summation processing operation by SIMD processing.



Fig. 12 Prototype 1.

setting of the area size and the number of captured images, output of about one image per 2 sec. could only be obtained as the processing time. Since the MAPP2200 had ability to capture and process binary images at 2,000 to 3,000 images per second, the reasons for the slow processing speed were thought to be: 1) it was necessary to conduct sequential comparison by providing a reference voltage 256 times when capturing an image at 8 bit resolution, but the column A/D could not provide individual reference voltage for each pixel, and 2) the architecture of the SIMD processor was specialized for binary images.

## 5 Second FS phase

Here, I shall explain the second FS phase for realizing Prototype 2 of the all-in-focus camera that allows real-time observation at 30 frames/sec..

In the microscope system, we were able to obtain cooperation of Photron Ltd., a company with abundant experience in high-speed image capture. In this system, the camerahead of the high-speed video camera was used, and by using LVDS that allowed high-speed image transfer as the interface between the imaging mechanism and the image processing arithmetic circuit, the imaging mechanism and the image processing arithmetic circuit were separated. The commercially available high-speed imaging mechanism (high-speed video camera) and the image processing arithmetic circuit (FPGA) were used. It can be said that the

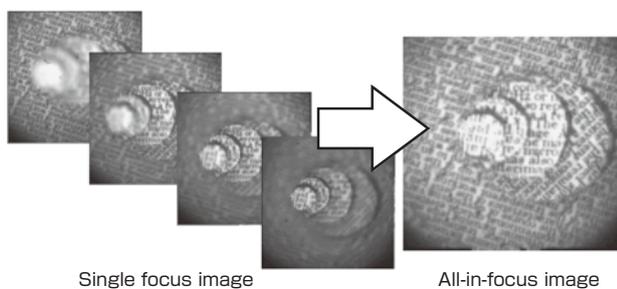


Fig. 13 Schematic diagram for the creation of all-in-focus image.

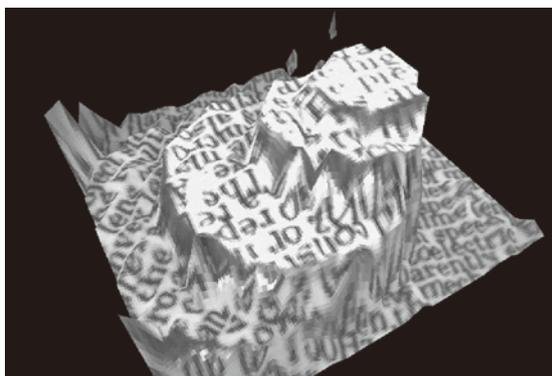


Fig. 14 Example of VR display.

road to product realization opened up widely by using the existing products.

The configuration of Prototype 2 is shown in Fig. 15 and the external appearance is shown in Fig. 16. The output of the high-speed sensor goes through the CDS (correlated double sampling) and ADC, converted by the high-speed digital interface LVDS, and then transferred to the image-processing unit. Up to this point is the description of the part of the high-speed camera. LVDS is a standard interface for sending high rate image signals, and is a standard often used in digital LC displays. The saw-tooth generating circuit for the varifocal lens receives the synchronizing signals from the clock generator of the high-speed camera part, produces saw-tooth pulses at 30 Hz, and drives the lens with the lens-driving amp. In the image processing part, the IQM calculation and image compositing are accomplished with the input digital image signals, and outputted as VGA (video graphics array) signals. The 3D data is transferred to the PC by LVDS signals. At the PC, data is received by the LVDS capture board of the PCI bus.

Mechanically, the focal distance movement used was the same as the one used in Prototype 1 and there was no problem. However, since the algorithm was implemented on the special vision chip in the first FS phase, it could not be transplanted directly to the FPGA, and it was necessary to modify the algorithm for FPGA. It was also necessary to use the internal memory to speed up the FPGA processing, and

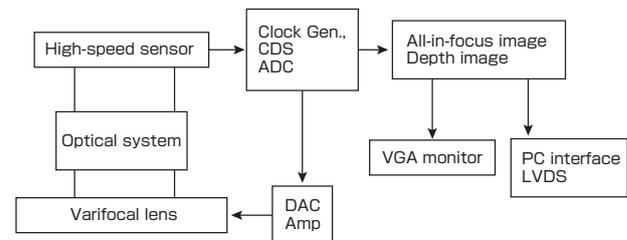


Fig. 15 Block diagram for Prototype 2.

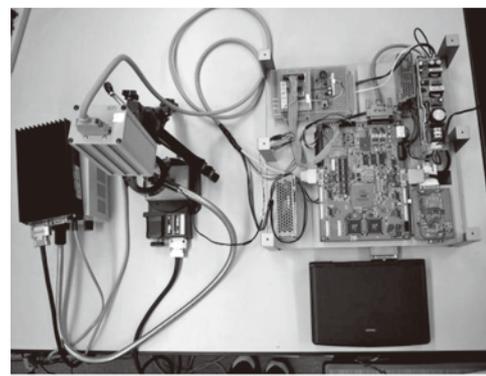


Fig. 16 External appearance of Prototype 2.

implementation to increase the memory capacity became necessary. Here, implementation of the sequential processing described earlier reduced the memory volume. As a result, processing could be accomplished within the internal memory of the FPGA, and target specification of 30 frames/sec. was achieved.

Figure 17 shows the examples of output images using Prototype 2. They look the same as the ones shown for Prototype 1, but actually, the all-in-focus images shown on the left and the depth images shown on the right are moving images. Eight focal points were recorded per frame, and as the performance of Prototype 1 was 0.5 frame/sec. while that of Prototype 2 was 30 frames/sec., the processing speed of the latter was 60 times faster.

### 6 Third FS phase

With the developments up to this point, the following points became apparent.

- By using the existing camerahead of high-speed cameras and LVDS, image capture, communication, and processing could be separated, and high-speed parallel processing using general FPGA was possible for the processing part.
- Mechanical focal distance movement could be done using varifocal lens.

From the talks with the company, while utilizing the high-speed processing part, we decided to specialize in the microscope use that had the highest potential in terms of business application. However, for the mechanical focal distance movement, the second item mentioned above, the varifocal lens could not be used due to precision issues. Here, I shall explain the development of the system specifically for microscope use in the third FS phase.

For the mechanism for moving the focal distance in the microscope system, we used the commercially available focal

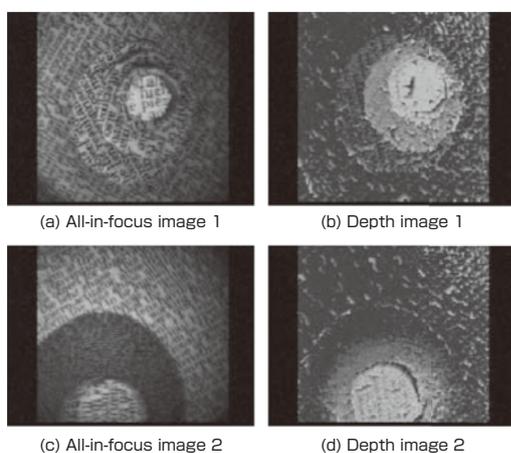


Fig. 17 Example of output by Prototype 2.

distance movement mechanism PZT actuator P-721 and 20 and driver E-612, C0 from PI Polytec Inc. By attaching these between the objective lens of the microscope, they enable parallel movement of the objective lens for 0-100  $\mu\text{m}$ . The reason for not using the varifocal lens used in the prototypes of chapters 4 and 5 was because in the varifocal lens, the  $f$  value of the lens itself was changed by changing the thickness of the lens. Therefore strictly speaking, images of different magnifications were composited, and this was not appropriate for producing high precision all-in-focus images.

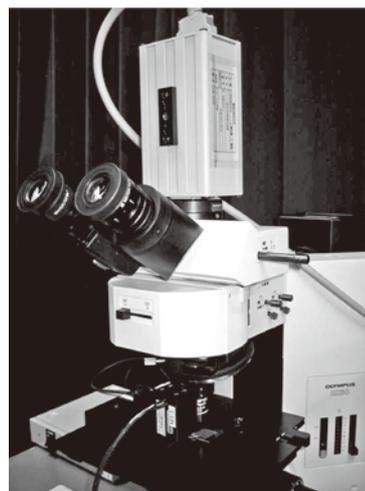


Fig. 18 External appearance of microscope system.

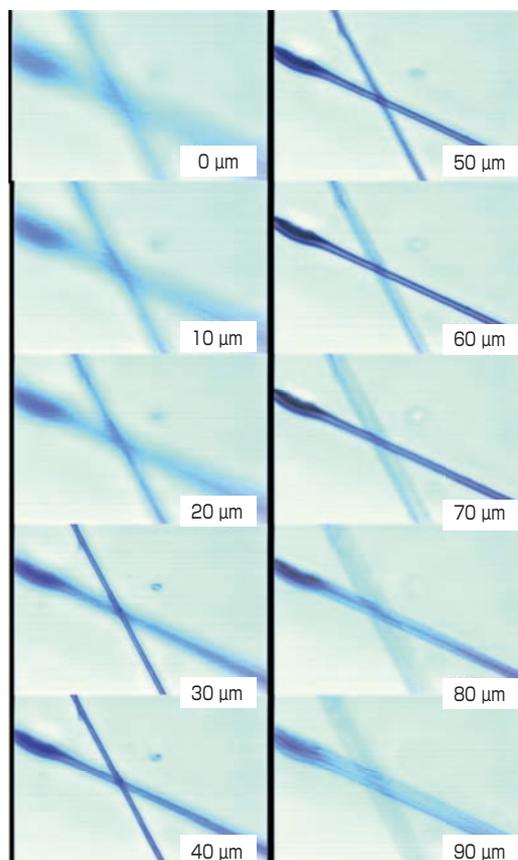


Fig. 19 Example of typical microscope image.

Figure 18 is an external appearance of the microscope system implemented on the microscope using Prototype 2 as a base. Figure 19 shows examples of ordinary optical microscope images, and Fig. 20 is an image obtained with the microscope system.

Here, glass fibers of 2  $\mu\text{m}$  diameter that are crossing each other three dimensionally are used as measured objects. Figure 19 shows the image every 10  $\mu\text{m}$ . It can be seen that the first glass fiber is in focus at 30-40  $\mu\text{m}$  while the second glass fiber is in focus at 60  $\mu\text{m}$ .

By using the proposed all-in-focus microscope camera, an all-in-focus microscope image, shown in Fig. 20, was obtained. Since this image is a movie, the all-in-focus image is sequentially updated even if the measured objects move. The depth image is also obtained.

## 7 Product phase

The system developed here was realized as a product by Photron in fiscal year 2003. The external appearance of this system is shown in Fig. 21. The basic configuration does not differ from the prototype, but measures are taken for practical use such as the incorporation of color images and keeping the size of the system as small as possible.

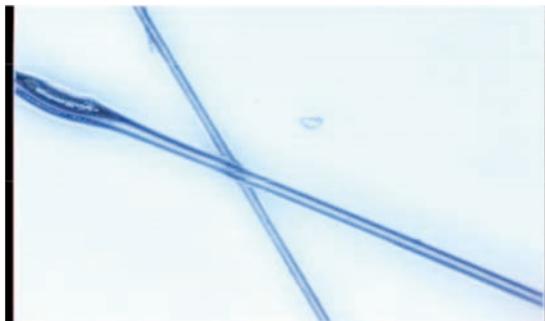


Fig. 20 Example of all-in-focus microscope image (moving image).



Fig. 21 External appearance of product system.

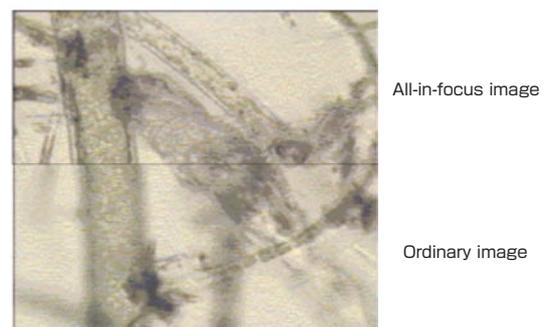
The examples of all-in-focus images obtained with this system are shown in Fig. 22, in comparison with ordinary images. Figure 22 top shows the wire bonding of an IC chip, and Fig. 22 bottom shows the diatom and microbes by transmitted light. For both images, the objective lens was of  $\times 50$  magnification. In actual performance, up to about  $\times 100$  objective lens can be operated, with the limiting factor being the weight of the objective lens. The stereomicroscope is used for low-power magnification from about 1 mm to 0.1 mm, and the electron microscope is widely used for high-power submicron observation. Due to the optical limit of the optical microscope, high-power is not needed in the biological field or for semiconductor inspection. Therefore, it was confirmed that the above magnification was sufficient for practical use.

## 8 Future issues

The all-in-focus microscope camera solves the issue of shallow depth of field that is a problem in ordinary microscope images, as well as the issue of not being able to obtain depth information. It is a system whose objective is to produce and display images where all areas are in focus and to conduct 3D compositions of objects in real time. By allowing all-in-focus images and 3D compositions at the same time, an object can be observed in detail. However, by specializing in extracting only the image in focus and the depth data from one direction only among the multiple images of varying focal distances obtained by ordinary microscope, much information had to be cut off. For example, when the focal distance is moved in a vertical direction for a



IC chip inspection



Microbe observation

Fig. 22 Example of image output of product.

translucent object, while there can be an object with several points in focus in the vertical direction, this method allows selection of one point only and unconditional observation of other areas become impossible.

As another method for reducing the optical scaling effect, the technology for volume rendering<sup>Term 4</sup> directly from the images with multiple focal distances, as shown in Fig. 23, was considered. This has been patented<sup>[9]</sup>.

In the future, it is expected that there will be demand for a hyper-microscope that can produce image slices from any direction by volume rendering all the depth images, without having to consider the hardware limitations.

## Acknowledgements

This study was based on the joint research by the Mechanical Engineering Laboratory, Agency of Industrial Science and Technology (current AIST), Ministry of International Trade and Industry; Delft High Tech Corporation (current DHT Corporation); Kawatetsu Techno Research Corporation (current JFE Techno Research Corporation); and Denso Corporation. The joint research for practical application was conducted by AIST and Photron Ltd. Part of the varifocal lens used in this study was manufactured by Denso Corporation, as a subcontract of the Micromachine Center that was subcontracted by the New Energy and Industrial Technology Development Organization (NEDO), as part of the “R&D for Micromachine Technology” based on the Industrial Science and Technology Frontier Program, Agency of Industrial Science and Technology, Ministry of International Trade and Industry.

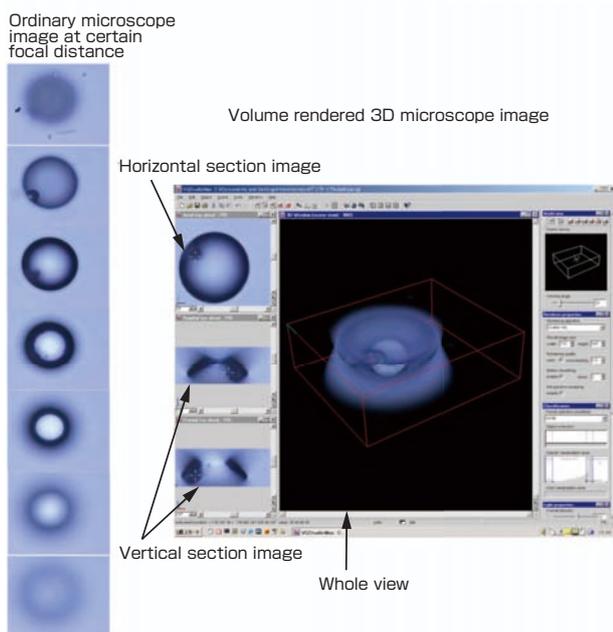


Fig. 23 Overview of volume rendering method.

## Terminology

- Term 1. LVDS (low voltage differential signaling) interface: standard of electric signal that enables high-speed function using twist pair cables. It has been used in high-speed cameras to handle high volume data. It is used in PCs as interface with LC displays.
- Term 2. FPGA (field programmable gate array) processing: a type of programmable gate array where the users can write their original logic circuit. The gate array is arranged in two-dimensional lattice form. It is suitable for parallel processing operation.
- Term 3. SIMD (single instruction multiple data) processing: process in an arithmetic device where simultaneous processing for several data is done with one command.
- Term 4. Volume rendering: When expressing a 3D object as a 2D image, rather than the method where the depth is expressed by adding shadow on the object surface, in this method, the 3D object is made to look three dimensional by adding transmittance and color information inside the object.

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## Discussion with Reviewers

### 1 Synthesis method

**Question and comment (Naoto Kobayashi, Center for Research Strategy, Waseda University)**

In this paper, the author has conducted an information configuration based on the original idea of the “configuration of real-time all-in-focus image” in the first part, then has conducted a system configuration including the hardware to realize the “configuration of real-time all-in-focus image,” and ultimately has achieved a product realization of the real-time all-in-focus microscope by clearly improving the objectives sequentially. This is extremely important. Here, we see that the extremely unique R&D unseen elsewhere, despite its twists and turns, has been conducted and completed according to the following steps: 1) consistency from *Type 1 Basic Research* to product realization, 2) sequential deepening and clarification of the strategic goals, 3) improvement of the required system configuration, and 4) actual product realization and maintenance as a commercial product thereafter. I thought these were appropriate as a *Synthesiology* paper.

On the other hand, in *Synthesiology*, the originality of the synthesis method is one of the key issues. For this paper, I guess that the strategy became clearer as the technology or the synthesis progressed, and the scenario became more apparent as it proceeded. I understand that there was no clear strategic objective such as “development and product realization of real-time all-in-focus microscope” in the beginning, but it fell into a cycle where the strategic goal deepened as the elemental technologies were synthesized, the issues to be solved in the next step became clear, and the next strategic goal deepened after the selection of elemental technologies and realization of the system. The elemental technologies were selected each time when you progressed to the next step, and the strategy became clarified and evolved during the progression. As a result, I assume that the methods of so-called “strategic deepening and selective synthesis” of elemental technologies were taken (see Figure a; modification of figure in *Synthesiology* 1(2) p.141). Is this view correct?

**Answer (Kohtaro Ohba)**

I think the method taken in the process of product realization could be called “strategic deepening” and “selective synthesis.” However, those were dependent on personal experiences and human contacts, and I feel it is extremely difficult to spell things out logically in a clean-cut manner to share it with others. I nevertheless have added that the feasibility study

phase was greatly affected by the “strategic deepening and selective synthesis”, including selections and rejections of some components, development of algorithms dedicated to those components, and meeting with partners to realize them, as well as the “lucky coincidences”. That is because as we blindly wandered in the dark and finally arrived at the goal, only in retrospect can we say certain places were the forks in the road. While I was groping desperately, honestly speaking, I cannot say I did any strategic decision-making even in retrospect. These points are discussed in “Chapter 2: History of twists and turns.”

### 2 “Valley of death”

**Comment (Hideyuki Nakashima, Future University Hakodate)**

There are the expression “valley of death” in several places. I think valley of death refers to the situation in which although there is technological prospect, other conditions (particularly cost) cannot be fulfilled toward product realization. It is a gap in research and development, where people do not want to get involved because it is not interesting as basic research since the principle is known, or because the development cost is too large for a company.

**Answer (Kohtaro Ohba)**

I think there is some difference in perception of the term “valley of death” as I understand it. The reviewer writes, “I think valley of death refers to the situation in which although there is some technological prospect, other conditions (particularly cost) cannot be fulfilled toward product realization,” but I don’t think it is the valley of death if one sees some technological prospect. Here, I use valley of death as a “place where one wanders in, loses the sense of direction of the goal much like the Forest of Aokigahara, and must make numerous twists and turns before arriving at the exit.”

**Comment (Hideyuki Nakashima)**

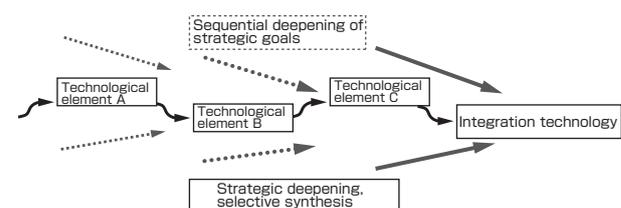
I think the part where the prospect of research cannot be seen (scene where a breakthrough is necessary) can be called the “wall,” “obstacle,” or “bottleneck.” I think you should refer to the “valley of death” in Wikipedia (Japanese version).

### 3 Synthetic explanation

**Comment (Hideyuki Nakashima)**

When we write an ordinary research paper, it is a presentation of a pathway taken in afterthought as we look back from the point the research is completed. We write as if the way to the conclusion was a straight path where we made all the choices without any doubt. The choices that were not selected or the accumulation of failures are not described in the paper. In that sense, the first manuscript was written like an ordinary paper.

Ordinary logical and analytical papers can have such structures. That is because the phenomena that must be understood are already in existence, and the purpose of a thesis is to present the pathway to the understanding. However, *Synthesiology* is for the discipline of synthesis. There may be more than one answer. The selection of certain paths amongst several possibilities itself is an important factor in synthesiology, and the descriptions of the selections are necessary. I think the content explained in the beginning of “Chapter 2: History of twists and turns” is important.



**Fig. a Structure of synthesis.**

# Portable national length standards designed and constructed using commercially available parts

— An advanced mechanical design for the iodine stabilized He-Ne laser —

Jun Ishikawa

[Translation from *Synthesiology*, Vol.2, No.4, p.276-287 (2009)]

The iodine stabilized He-Ne laser at 633 nm is widely used as the national length (wavelength) standards in many countries. Since the wavelength emitted by the laser is directly proportional to the cavity length of the laser, extremely high mechanical stability is necessary for the cavity of the iodine stabilized He-Ne laser. Many special parts as well as special materials are adopted to achieve a sufficiently high stability in the conventional iodine stabilized He-Ne lasers while the adoption of such special parts and materials brings difficulty in the maintenance of the lasers. I developed and constructed a new iodine stabilized He-Ne laser with a special mechanical design. The assembly and adjustment of the laser is quite easy. Although most parts and materials of the laser are commercially available, it showed better stability especially against the ambient temperature variation. The new iodine stabilized He-Ne lasers were used for a long time as the national length standards of Japan.

**Keywords :** Mechanical design, fine mechanism, anti-vibration mechanism, length standard, iodine stabilized He-Ne laser

## 1 Introduction

The length is one of the most basic physical quantities, and the technology for measuring the length is highly advanced in the fields of science and industry. The international unit for length is the meter (or metre). The international prototype of "X" cross-section metre line-standard was used as the standard since the latter half of the 19th Century, and in the 20th Century, a definition of the metre in terms of the wavelength in vacuum of the radiation corresponding to a transition between specified energy levels of the krypton 86 atom was adopted in 1960. The current definition of the metre has been adopted since 1983, and this is based on the speed of light traveling in vacuum (light velocity)<sup>Note 1)</sup>. Specifically, the standard is the wavelength of laser radiation under specific conditions. The most widely used standard is the helium-neon laser with its laser wavelength stabilized to the absorption line of iodine molecules<sup>Note 2)</sup>. Currently many countries around the world including Japan use the iodine-stabilized He-Ne laser as their national standards. Figure 1 is the traceability system of length measurement using this laser as the national standard.

In general, the national standard is expected to possess the highest precision above the measurement precision required in industry and academia. Therefore, the latest, most advanced, and special technologies are summoned to achieve the highest precision in the development of the national standard. When the R&D is completed and the national standard is created, the next phase is how the standard is supplied effectively to industry and academia, and how to maintain this national standard stably. Once this supply phase is

reached, the latest, most advanced, and special technologies become barriers in supplying and maintaining the standard due to high cost and difficult availability, which are factors that cannot be neglected.

When the author started research on precision interferometry at the former National Research Laboratory for Metrology, Agency of Industrial Science and Technology (current National Metrology Institute of Japan, AIST), the iodine-stabilized He-Ne laser used as wavelength standard (this was national standard of length in Japan to 2009) faced such problems. In the beginning, although I experienced poor operability and problems in procuring the special parts, I thought they were unavoidable hardships for the upkeep of a national standard with highest precision. At that time, a researcher of Physics and Engineering Laboratory of New Zealand (the National Standards Laboratory) was visiting the standard labs of various countries and stopped at AIST to conduct an international comparison of iodine stabilized He-Ne lasers<sup>[1]</sup>. He hand-carried a self-developed laser onto the passenger cabin of the airplane. Compared to the Japanese laser that was composed of a rack full of control components and the laser body installed on a cast-iron channel bench, I was absolutely amazed by its compact size and easy operability.

Inspired by this experience, I started research on the universalization of the iodine stabilized He-Ne laser. Here, "universalization" means to carefully study and to clarify the functions and properties required for each member of the iodine stabilized He-Ne laser, and then to achieve these functions and properties using generally available parts as much as possible. In this paper, I shall describe the

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situation when I started the research, the policy for elemental technology development taken under that condition, what kind of elemental technologies were developed, and what national standard was achieved by combining the elemental technologies.

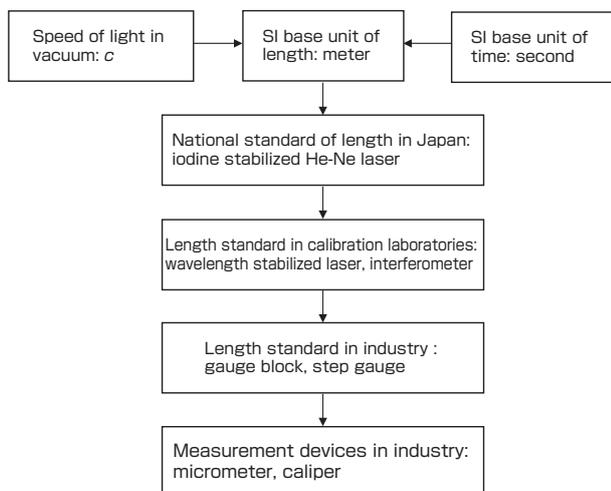
## 2 Objective of research

### 2.1 Universalized national standard

The specifications and performances required for any standard, including the iodine stabilized He-Ne laser, and its member parts may be special compared to general products. In the basic development phase, because the priority is to realize the required performance, special and expensive materials, parts, and processing methods are used generously.

In the beginning, the author was not in the position to develop the standard, but was involved in the iodine stabilized He-Ne laser as a user, and came across the opportunity of seeing the New Zealand laser. From that experience, I was convinced that the basic development phase was only half of the entire course of development of the standard, and universalization was essential to accomplish the development completely. As mentioned earlier, universalization is to carefully study and to clarify the performances and properties required and to establish the design and technology that achieve those performances and properties using universally available parts. I consider universalization as an attempt at conversion from special parts (hardware) to design and technology (software).

The operating principle of the iodine stabilized He-Ne laser is based on the quantum mechanical property of the iodine molecule. Since the quantum mechanical property of the iodine molecule is consistent, there is no difference in principle among the iodines used, unlike the International Prototype of metre that uses artifacts. However, flaws arise in



**Fig. 1 Traceability system of the length measurement using the iodine stabilized He-Ne laser as the national standard.**

the mechanism, which are artificial, for extracting the stable laser wavelength based on the quantum mechanical property of the iodine molecule, and this manifests as differences and uncertainties in laser wavelengths by different devices. The author thinks that possession and maintenance of the standard is not merely possessing the iodine stabilized He-Ne laser (hardware), but is the possession of the technology to extract the laser wavelength from the iodine molecule. For universalization, careful investigation and deep understanding of the laser wavelength extraction are necessary. The possession of technology gained through universalization is none other than the possession and maintenance of the standard.

It is expected that there will be many advantages in universalizing the national standard. The main advantages are the increased mutual reliability of the length standard among countries, and dramatic improvement of the precision of fine measurement of length in Japan.

### 2.2 Increased international mutual reliability

Except for some of the advanced nations, most national standard labs purchase commercially available iodine stabilized He-Ne laser as their national standard. In such cases, they must depend on the laser manufacturer for repairs other than simple adjustments. In other words, the national standard labs simply possess the hardware, and the maintenance of the standard is entirely dependent on the manufacturer. There will be no major problem as long as the product has a high degree of perfection and there are no delays in supply and maintenance services. However, because a national standard device like the iodine stabilized He-Ne laser requires high technology and its demand is extremely limited, it is not easy to develop, manufacture, and maintain it as profitable business. In fact, the business is fraught with difficulties.

The author considers the universalization of iodine stabilized He-Ne laser to be the realization of a laser with excellent performance, operability, economy, and ease of maintenance as a standard, and at the same time to disclose the detailed technological information to the laser users. The essence of wavelength standard is the technology to obtain stable laser wavelength from the unchanging quantum mechanical property of the iodine molecule. If the technology can be shared among the countries through universalization, it can become the foundation for establishing the mutual reliability to confirm the equivalencies of the national standards among the nations.

### 2.3 Advancement of mid-tier traceability in Japan

There are several companies that provide calibration service for wavelength standard as business in the middle tier of traceability in Japan, and their technological level is almost par to the national standard labs except for some advanced nations. The employment of the universalized iodine

stabilized He-Ne laser as the standard of the calibration service providers means the possession of the standard as a technology, as explained in the previous section.

The traceability of artificial standards such as gauge blocks and weights can be maintained by assessment through calibration. However, the iodine stabilized He-Ne laser is a standard that extracts wavelength based on the quantum mechanical property of the iodine molecule, and the difference by device (uncertainty) depends on the technology for extracting the wavelength. In such a standard, in addition to evaluating the wavelength, the possession of technology for extracting the wavelength and its assessment, or the “traceability of technology” is essential. Universalization of the technology will promote conversion from possession of hardware to possession of technology, and the reliability in the mid-tier traceability such as among the calibration service providers will improve.

### 3 Operating principle of iodine stabilized He-Ne laser

Here, I shall explain the operating principle of the iodine stabilized He-Ne laser. Figure 2 is a schematic diagram of the structure of the iodine stabilized He-Ne laser. In ordinary He-Ne laser, a laser tube containing a mixture of helium and neon gases is placed between the laser cavity composed of two plane laser mirrors (more accurately, they are slightly concave). In the iodine stabilized He-Ne laser, the “iodine cell” where highly pure iodine molecule is sealed inside is placed in the laser cavity. The relationship between the optical interval (length of laser cavity) of the two laser mirrors  $L$  and wavelength  $\lambda$  can be expressed as Equation (1).

$$\lambda = 2L / N \quad (1)$$

Here,  $N$  is an integer.  $\lambda$  is proportional to  $L$ . Since the wavelength range in which the light amplification effect of the He-Ne laser tube is effective is extremely narrow, the laser wavelength  $\lambda$  remains at a certain limited range. When  $L$  is changed past the effective wavelength range, the integer  $N$  increases or decreases one at a time (mode hop), and remains at a certain range in which the light amplification is effective. When  $L$  is varied in the range where mode hop does not occur, the laser output  $I$  changes as shown in Fig. 3 (dashed line).  $I$  decreases at both ends where  $\lambda$  is close to mode

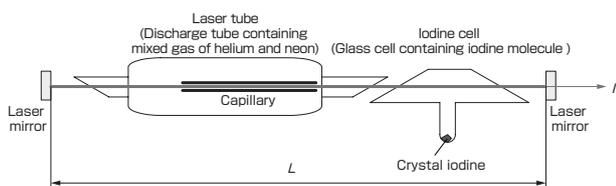


Fig. 2 Schematic diagram of iodine stabilized He-Ne laser.

hopping, and becomes highest at the center. When the iodine cell is present in the laser cavity in addition to the laser tube, the light absorption by the iodine molecules affects  $I$ , and the output curve changes as shown in Fig. 3 (solid line). Since a strong standing wave of light (10 mW) is present inside the laser resonator, the absorption by iodine molecules becomes saturated. Since the absorption weakens at the center of the absorption wavelength by saturated absorption,  $I$  increases slightly, and a spike appears on the output curve. Using this phenomenon, extremely high-resolution spectroscopy (saturated absorption spectroscopy) without the Doppler effect due to motion of the iodine molecules can be achieved. The iodine stabilized He-Ne laser uses the spike of saturated absorption that appears on the output curve as a marker, and the high precision is realized by controlling and stabilizing the laser wavelength to its center.

Differential signal by phase sensitive detection is normally used as method for detecting the peak position of the output curve. Figure 4 shows the principle of differential signal by phase sensitive detection. To accomplish the differential signal,  $\lambda$  is slightly modulated. The modulation of  $\lambda$  changes the  $I$ , but the amplitude and phase are determined by the gradient of output curve  $I(\lambda)$ . As shown in the figure, the first derivative  $I'(\lambda)$  of  $I(\lambda)$  is obtained when the DC component is extracted from the signal obtained by crossing the output signal and the demodulated signals. The laser wavelength stays at the peak position of  $I(\lambda)$  by controlling the  $\lambda$  to keep this derivative at zero. However, in the iodine stabilized He-Ne laser, since the spike due to saturated absorption of iodine molecules is superimposed on the laser output curve  $I(\lambda)$ , the effect on the gradient of the baseline cannot be avoided. To remove this effect and to detect the true center of the saturated absorption by iodine molecules, the third derivative detection is done in practice. The signal  $I'''(\lambda)$  of the third derivative can be obtained by using the threefold wave (frequency  $3f$ ) of the modulated signal (frequency  $f$ ) as the demodulated reference signal of the phase sensitive detection. Since the laser output curve is gentler by far compared to the spike of saturated absorption, the effect is sufficiently removed by third derivative. Figure 5 is the

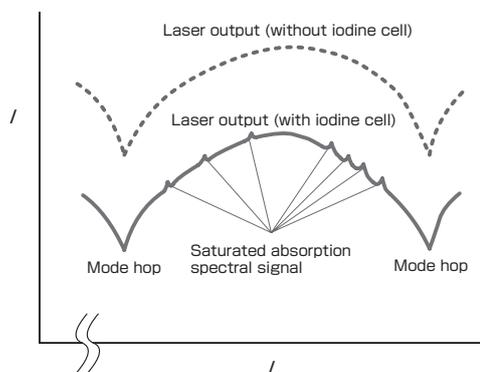
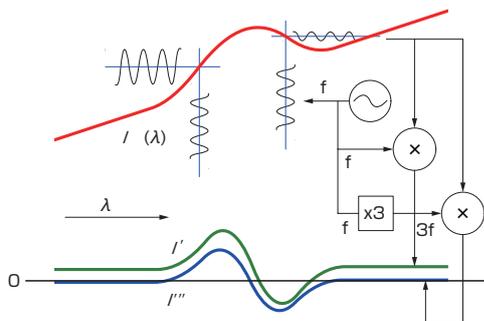


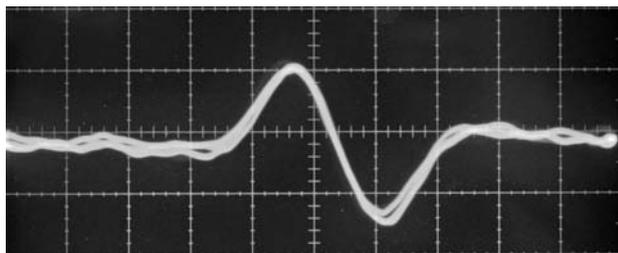
Fig. 3 Relationship between laser cavity length  $L$  and laser output  $I$  (in single axial mode).

third derivative signal  $I'''(\lambda)$  around the iodine molecule absorption component in the iodine stabilized He-Ne laser output observed on the oscilloscope. The laser wavelength is controlled so this third derivative signal becomes zero and stability is achieved.

The wavelength of laser  $\lambda$  is proportional to the length of the laser cavity  $L$  in the range where mode hop does not occur, as mentioned earlier. Therefore, to change the laser wavelength  $\lambda$  for control and modulation,  $L$  may be changed. For example, 6 MHz<sub>p-p</sub> modulation, converted to optical frequency, is applied to the laser wavelength  $\lambda$  as modulation for third derivative signal detection. If  $L$  is 0.3 m, the change of  $L$  corresponding to the 6 MHz modulation is 3.8 nm. This means that if one of the laser mirrors is vibrated at amplitude 3.8 nm in the optical axis direction, modulation of  $\lambda$  needed for third derivative detection can be achieved. Also, the width of the third derivative signal (Fig. 5) used in the control of laser wavelength is about 5 MHz, converted to optical frequency. When the modulation range of the laser wavelength surpasses this width due to vibration or shock, control is lost (this is when “the frequency lock becomes unlocked”). To stabilize the laser wavelength, it is necessary to keep the change in  $L$  at about 1/10 of this width, or at about 500 kHz. The change of resonator length  $L$  equivalent to the change of laser frequency change of 500 kHz is 0.32 nm. This is approximately the diameter of an atom. For a stable operation of the iodine stabilized He-Ne laser, it is necessary to have the fine technology of keeping the change in the interval of the laser mirror due to vibration to about the diameter of an atom.



**Fig. 4 Spike in laser output  $I(\lambda)$  due to saturated absorption, signal for its first derivative  $I'$ , and signal for third derivative  $I'''$ .**



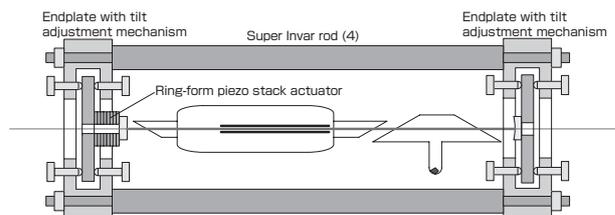
**Fig. 5 Actual third derivative signal observed on the oscilloscope.**

Figure 6 is the mechanism of the first resonator of the iodine stabilized He-Ne laser used by the author. One of the laser mirrors is as is, while the other one is attached to a ring-shaped stacked piezoactuator. Each mirror is supported by endplates with mechanism for precise angle adjustment. The two endplates are supported facing each other with four spacer rods made of Super Invar that has thermal expansion close to zero. A laser tube and an iodine cell are set in the resonator. In actual operation, the effect of vibration was kept within the aforementioned condition range of 500 kHz by mounting the resonator on the anti-vibration plate supported by air springs and then covering it with a soundproof case. Only one multi-layer ring piezoactuator is shown in Fig. 6, and both the demodulation and the control signals were applied to this piezoactuator to accomplish simultaneous modulation and control.

#### 4 Road map for achieving the goals

As mentioned in the earlier chapters, the performances and properties required from each mechanism of the iodine stabilized He-Ne laser are quite special. Special requirements cannot be achieved by using universal members. To achieve special performances and properties using universal parts, it is necessary to devise new and special usage. As for special usage, methods that cause danger or ones that significantly reduce the lifespan cannot be employed. By devising and realizing ways to achieve the required performances and properties within reasonable and feasible range, the employability of the universal parts can be widened even if they are used unconventionally.

As an example, I shall explain the new bias adjustment circuit of the control mechanism. In the iodine stabilized He-Ne laser, it is necessary to smoothly change the voltage applied to the piezoactuator to search and select the absorption line. Conventionally, this change was done using the potentiometer, but there is no potentiometer that has sufficient voltage resolution over the entire range. Therefore, the range of change was limited to a narrow voltage range, and this made the operability poor. Figure 7 shows the voltage adjustment circuit that achieves extremely high voltage resolution using the DC gear motor and integrator circuit instead of a potentiometer. The DC gear motor is an actuator that rotates at rate proportional to the input



**Fig. 6 Resonator mechanism of the iodine stabilized He-Ne laser (conventional type).**

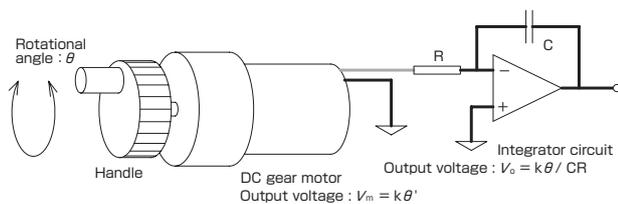
voltage, but when it is rotated by external force, it also functions as a generator and produces voltage proportional to the angular velocity ( $2\pi \times$  number of rotations). When the generated voltage is input to the integrator circuit, the output is voltage proportional to the rotational angle of the gear motor. The constant of proportion of the rotational angle and the integrator circuit output voltage can be set arbitrarily by selecting the input resistance of the integrator circuit and the capacitor. Moreover, this gear motor can be rotated without limit, unlike a potentiometer, and the voltage can be adjusted throughout the process. For example, it can be set at a rotation of 100 times or more.

Achievement of special functions using universal parts was not conducted in a planned or systematic manner. While the development of parts directly affecting the laser performance as well as parts that may be difficult to procure or to maintain should be priority, the order at which ideas arose and problems were solved did not necessarily correspond to the priorities. Therefore, in conducting this development, there was no planned or systematic scenario in the beginning. After I succeeded at several universalizations, I became capable of seeing the bottleneck accurately. By recognizing the importance of the issue and then placing it somewhere in the corner of my mind, I was able to engage in improvements and developments whenever a solution came up while doing some other work. Under such situations, the set of universalizations was completed in about 10 years.

The example of the above voltage adjustment circuit improved the operability, and it was possible to improve the basic performance (uncertainty and stability) by using the new mechanism with universal parts. The following chapters explain the control mechanism of the laser cavity length that determines the basic performance of the laser.

### 5 Universalization of laser cavity length control mechanism

The laser resonator length control mechanism is the most important part that determines the basic performance of the iodine stabilized He-Ne laser. Conventionally, special parts such as ultra-low heat expansion materials and piezoactuators were used. The resonator length control mechanism is composed of the linear movement mechanism to control the wavelength and the modulation mechanism to obtain



**Fig. 7 Wide-range voltage micro-adjustment circuit using DC gear motor and integrator circuit.**

**Table 1 Operational parameters of the iodine stabilized He-Ne laser (recommendation of the CIPM).**

· Cell-wall temperature of iodine cell	(25±5)	°C
· Cold-finger temperature of iodine cell	(15±0.2)	°C
· Frequency modulation width peak-to-peak (P-P)	(6±0.3)	MHz
· One-way intracavity beam power	(10±5)	mW
* The coefficient of one-way intracavity beam power of the optical frequency must be 1.4 kHz/mW or less.		

the third derivative signal. The universalization of the two mechanisms is explained below. Also, I shall explain the anti-vibration mechanism that is important to achieve stable resonator length control.

#### 5.1 Linear movement mechanism of the laser mirror

The operating parameters of the iodine stabilized He-Ne laser are listed as the recommendation of the Comité International des Poids et Mesures (CIPM)<sup>[2]</sup>. Table 1 shows the operating parameters of the iodine stabilized He-Ne laser as recommended by CIPM. Of the four recommended parameters, only the depth of the modulation ( $6 \pm 0.3$  MHz converted to optical frequency) as mentioned earlier is directly related to the resonator mechanism, but since the one-directional optical intensity ( $10 \pm 5$  mW) within the resonator is affected by the laser mirror angle, it can also be considered as an issue of the resonator mechanism. In the conventional iodine stabilized He-Ne laser, the resonator length was controlled by a ring-shaped stacked piezoactuator. However, the piezoelement is not necessarily made of homogenous materials, and unevenness where the expansion and contraction may differ in places may occur even when constant voltage is applied. This causes a phenomenon where the tilt angle of the laser mirror slightly varies. When the tilt of the laser mirror changes, the loss of laser cavity changes so that the laser output also changes. The laser output is determined by the sum of the one-directional optical intensity within the resonator and the transmissivity of the laser mirror. Table 1 shows that the tolerance of change is large at  $\pm 50\%$  of the designated output (one-directional optical intensity in the resonator is 10 mW), and the output change due to change in the tilt of the piezoactuator normally falls within this range.

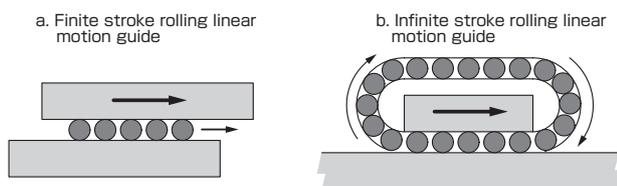
However, from actual experiments, it was absolutely necessary to keep the change of the tilt of the laser mirror during control as small as possible, to realize the iodine stabilized He-Ne laser with excellent reproducibility and stability of laser wavelength. On this point, the recommendation of CIPM is deficient on the major parameter that affects the laser wavelength. The author has conducted measurements while maintaining the laser power  $I$  almost constant while changing the angle of the laser mirror, to assess the dependency of the laser wavelength  $\lambda$  on the inclination of the laser mirror<sup>[3]</sup>. As a result, it was experimentally confirmed that the laser wavelength may

change in the recommended uncertainty (10 kHz converted to frequency), even when all the operating parameters of the iodine stabilized He-Ne laser are set within the recommendations of CIPM. It was learned that extremely high linearity was required for the movement mechanism of the laser mirror to ensure the stability of laser wavelength of the iodine stabilized He-Ne laser. The piezoactuator used in the control of the resonator length has excellent rigidity and micro-displacement capability, but was a bottleneck for the precision of conventional iodine stabilized He-Ne laser due to the linearity issue. I learned that it was essential to develop a movement mechanism with excellent linearity for further universalization, and employed the method of combining the piezoactuator with excellent rigidity and micro-displacement and a guide mechanism with excellent linearity in the new iodine stabilized He-Ne laser.

In general, the mechanical linear guide can be categorized into three: parallel spring, sliding, and rolling guides. The parallel spring guide has been used most commonly as the linear guide combined with the piezoactuator. Although the range of motion is limited, the parallel spring guide is considered particularly suitable for fine position control since there are no allowance, friction, or backlash. However, to realize a high degree of linearity, the parallel spring structure must be somewhat enlarged, and the weight increases when the rigidity of the system increases. Moreover, it is expensive. The sliding guide inherently has allowance and cannot be used for this purpose. In this development, I use the rolling guide because it is inexpensive and light, even though it had almost no history of being used as a guide for laser cavity length control.

### 5.1.1 Linear motion guide by ball spline

Rolling linear motion guide includes ones with finite stroke and infinite stroke. The finite stroke rolling guide has structure where the rolling body such as balls or rollers is placed between the opposing linear guides, as shown in Fig. 8a. When the lower guide is fixed and the upper guide is moved, the rolling body in between moves halfway along the upper guide. To maintain the movement of the guide, the rolling body is arranged at the range of 50~70 % of the total length of the guide. The problem of the finite stroke rolling guide is that the support point (position of the rolling body) moves along with the movement. While it is dependent on the rigidity and load, the movement of the support point is the cause of unavoidable position shift.

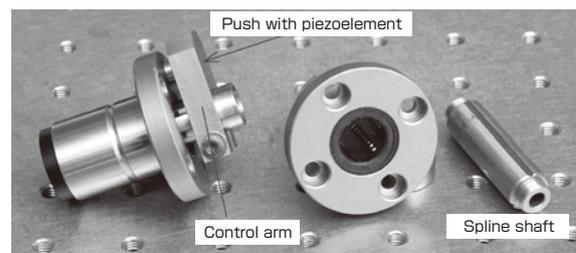


**Fig. 8 Finite stroke rolling linear motion guide (a) and infinite stroke rolling linear motion guide (b).**

The infinite stroke rolling guide has a mechanism to return the rolling body, which is expelled from the rear as the upper guide moves, to the position between the upper and lower guide, as shown in Fig. 8b. In addition to allowing long distance movement, the movement of the support point is small (less than the interval of the rolling bodies), and it is an excellent mechanism with little position shift.

The first mechanism I used as the rolling linear motion guide was a ball spline with a circular hollow shaft structure (THK Co., Ltd.: LF13)<sup>[4]</sup>. The ball spline is a kind of infinite stroke rolling guide, and I decided to use this because high-level linearity can be obtained. Also it was shaped similarly to the ring-form piezo stack actuator and it could be incorporated easily to the laser resonator. The spline shaft diameter of the ball spline is 13 mm, and there is a hole of 5 mm diameter in the center through which the output laser passes. Figure 9 shows the laser mirror linear movement mechanism with an incorporated ball spline. The laser mirror is set at the end of the spline, and the spline shaft is pulled out front with a preload spring in the control arm. When the adjustment mechanism for the laser mirror is installed on the laser body, the tip of the linear piezoactuator of the main body contacts the spline shaft control arm, and the spline shaft is pushed in about halfway. Control voltage is applied to the linear piezoactuator, and the mirror position is controlled by the expansion and contraction.

Unlike the sliding guide, the allowance can be removed in the rolling mechanism by applying preload. For the ball spline tested, the gap between the main body and the ball spline shaft was adjusted to less than the ball diameter by 2 ~ 6 μm, and there was no looseness. However, when the spline shaft was pushed in the side direction during laser emission, a change in laser power that was thought to arise from the mirror tilt was observed. The rigidity against the side moment of the ball spline guide is not enough. In this ball spline guide, the ball is circulated. According to the specification, since the gap between the main body and the spline shaft is smaller than the diameter of the ball (-6 ~ -2 μm), large resistance is produced when the ball enters the interval and hampers smooth movement. However, no change in resistance due to entry and exit of the ball was observed. In both ends, the interval is slightly larger to prevent resistance when the ball enters, and it is estimated that the actual



**Fig. 9 Linear motion guide for laser mirror with the ball spline.**

preload occurs in the central part. As a result, the rigidity against the shift can be maintained but the rigidity against the tilt is insufficient. In the manufacturer's explanation, it is recommended that two splines should be used linearly in place where there is side moment.

### 5.1.2 Linear movement mechanism by cross roller guide

It was mentioned earlier that the position shift accompanying the movement of the support point position couldn't be avoided in the finite stroke rolling linear motion guide. In principle, this position shift is thought to gradually progress throughout the stroke. On the other hand, the stroke needed for stabilizing the laser wavelength is around 0.05 mm since the purpose is to correct the expansion and contraction of the laser cavity length due to heat expansion. This is 1/100 or less of the maximum stroke of the cross roller guide<sup>[5]</sup>, which is a representative limited distance rolling guide, as well as for the smaller versions. For the position shift for the entire stroke of the cross roller guide, the position shift against the movement of 1/100 stroke is expected to be sufficiently small, while it may not fulfill the performance required for the linear mechanism of the iodine stabilized He-Ne laser. Moreover, in the finite stroke rolling guide, since there is no entry or exit of the rolling body into the guide, it is not necessary to widen the ends of the guide as in the infinite stroke rolling guide. Therefore, it is expected to have high rigidity against tilt. After considering the conditions and required performances of the linear motion guide for cavity length control, as well as the performance of the linear motion guide used under that condition, cross roller guide emerged as an optimal linear motion guide.

The shape of the cross roller guide is suitable for a movement stage, and application to circular mechanism is difficult. With employment of the cross roller guide, the structure of the laser cavity was greatly changed from the conventional endplate and rod to a housing combining the plate members as shown in Fig. 10. Moreover, making use of the separation of the movement control of the laser mirror and the maintenance of linearity, I devised a structure where the

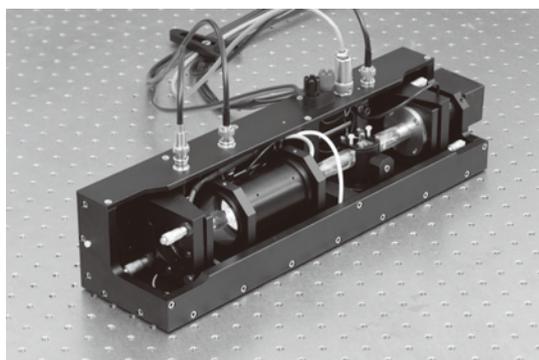


Fig. 10 Box-shaped laser cavity mechanism of the iodine stabilized He-Ne laser (new type).

linear piezoactuator was fixed with the Super Invar rod, and the other side of the Super Invar rod was fixed to the housing at the position of the other laser mirror, as shown in Fig. 11. With this structure, the resonator length is determined by the piezoactuator and the Super Invar rod. Since it is not necessary to consider the heat expansion of the housing material, aluminum could be used. Since aluminum has high heat expansion, it was not conventionally used as the housing of the laser resonator. However, it also has high heat conductivity, and it has excellent characteristic where the heat strain due to heat gradient is not likely to occur. Compared to Super Invar rod, the heat expansion coefficient of aluminum is 50 times more, but the heat conductivity coefficient is also close to 20 times, and the effect of the heat strain will remain within 3 times.

The new laser with the cross roller guide, aluminum housing, and control mechanism for resonator length by linear piezoactuator and Super Invar rod has extremely small tilt angle change of the laser mirror. Stable operation with small temporal change and environmental dependency has been achieved. Currently, among the five new lasers owned as the national standard of Japan, the difference in laser wavelength is held within  $\pm 3$  kHz converted to optical frequency by conducting appropriate adjustment. This is within 1/3 of the  $\pm 10$  kHz uncertainty recommended by CIPM. It is also stable against the changes in environmental temperature, and shows excellent performance where the change of the laser wavelength stays within the deviation of  $\pm 5$  kHz converted to optical frequency (relative  $1 \times 10^{-11}$ ) even against large temperature shift of  $25 \pm 5$  °C.

### 5.2 Modulation mechanism of the laser resonator

In the iodine stabilized He-Ne laser, modulation of  $6 \pm 0.3$  MHz converted to optical frequency is applied to laser wavelength to detect the saturated absorption signal of the iodine molecule. This modulation is accomplished by vibrating one of the laser mirrors by sine wave in the optical axis direction. In the case where the resonator length is 30 cm, the amplitude is  $(3.81 \pm 0.19)$  nm. As mentioned

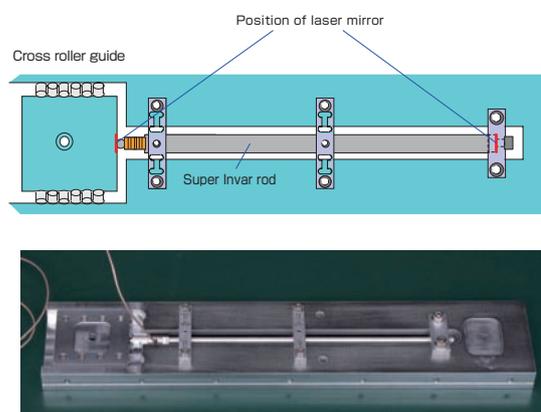


Fig. 11 Control mechanism for cavity length.

earlier, to obtain the control signal by third derivative, the third harmonic detection is done. When the third harmonic is included in the vibration of the laser mirror, the first derivative signal component enters into the third derivative signal, and the laser wavelength shifts as a result. Therefore, the vibration of the laser mirror is required to be pure fundamental with extremely low harmonic distortion. Moreover, to maintain the sensitivity of the phase sensitive detection constant, the phase relationship of the driving sine wave and the vibration must not change. Extremely high level of amplitude stability, ultra low distortion, and phase stability are required for the wavelength modulation actuator. In many iodine stabilized He-Ne lasers including the conventional laser, ring-form piezo stack actuator with the same structure as the ones to control the resonator length are used for modulation. In the conventional laser, one ring-form piezo stack actuator was used for both modulation and control.

The piezoactuator with stacked or multilayer structure is originally for control (DC operation), and the use with vibration (AC operation) is not assumed. The durability when it is vibrated is an issue. In conventional lasers, detachment of the adhesive surface of the piezoelement caused by modulating vibration occurred frequently. When detachment occurred, it cannot be repaired and the entire component must be replaced. The US made ring-form piezo stack actuator was expensive and the supply was unstable (because they were prioritized for military use), and this was a major barrier in using it in the iodine stabilized He-Ne laser.

### 5.2.1 Modulation mechanism using single-layer piezoelement

The detached ring-form piezo stack actuator must be discarded. One day, I attempted disassembling it to understand how it failed. By soaking the stacked piezoactuator in acetone, all the adhesives detached and it came apart easily. I found out that the stacked piezoactuator was constructed by stacking 10 piezoelement discs to which metal was deposited on both sides to function as electrode. Since the stroke of the actuator is 10  $\mu\text{m}$  for applied voltage 1000 V, one element is 1/10 or 1  $\mu\text{m}$ .

With the dissembled actuator in front of me, I thought of developing a single-layer modulation actuator using one element. There will be no detachment if it has only one layer. The amplitude necessary for modulation is about 4 nm as mentioned earlier, and the applied voltage equivalent to the displacement of the single-layer element is 4 V, a voltage that can be handled readily by circuit using ordinary op amp. Moreover, since the rigidity will increase greatly, the stability of operation can be expected to improve.

Figure 12 is the structure of the modulation actuator that was first fabricated. It has a simple structure where a ring-shaped piezoelement, an aluminum laser mirror holder,

and an O-ring were held down in a case between two brass securing ring screws. The voltage is applied between the laser mirror holder and the securing ring. However, when this mechanism was set in the iodine stabilized He-Ne laser and I attempted stabilizing the wavelength, large wavelength shift over 10 times the recommended uncertainty was observed. The baseline of the third derivative signal was so tilted that it could be discerned visually, and there was an inclusion of the first derivative signal, or the substantial occurrence of third harmonic distortion in the modulation mechanism. In this structure, when static, the soft O-ring distorted and functions as expected. However, in vibration, the brass securing ring supporting the piezo vibrates due to action and reaction. The brass securing ring is in contact with the main body by the thread of the screw. The contact points of ordinary screws are partial, and it is thought that the rigidity is low against small displacement and nonlinearity is high. I suspected that the structure where the screws directly supported the object caused distortion.

### 5.2.2 Improvement of dynamic property of the modulation mechanism

To improve the dynamic property and to reduce the harmonic distortion of the modulation mechanism, it is essential that the mechanical contacts are carefully finished planes to maintain highly rigid and stable contact. Moreover, it is important that the holders that support the vibrating laser mirror are immobile as much as possible. Considering these two points, Fig. 13 shows the modulation actuator that reduces the harmonic distortion and improves the operational stability. The point that differs greatly with the modulation actuator in Fig. 12 is the mass of the brass support base. The mass of the brass support base is about 200 g, and is about 50 times the mass of the 4 g vibrating part including the laser mirror and the holder. Due to this large mass ratio, only the laser mirror and the holder move even if the ring-shaped piezoelement expands and contracts at high speed, and the brass base remains immobile. Particular care is taken for

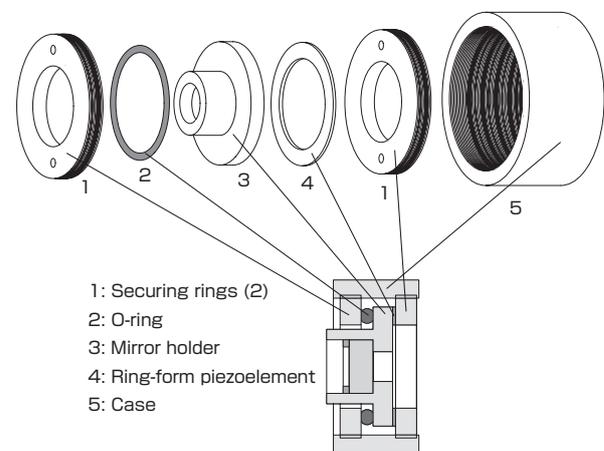


Fig. 12 Modulation actuator (before improvement, large harmonic distortion).

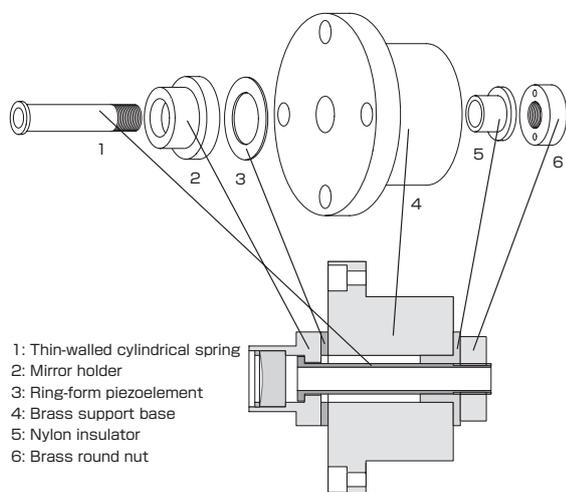
the finish of the contact planes of the brass support base, the ring-shaped piezoelement, the laser mirror holder, and the laser mirror. Lapping finish is done to increase the contact surface area to reduce the harmonic distortion. Also, the ring-shaped piezoelement and the laser mirror holder are placed on the brass support before assembly. Alcohol is poured on the contact plane, the brass support is flipped over, and firm planar contact is confirmed by seeing that the ring-shaped piezoelement and the mirror holder are fast and do not fall off.

The mirror holder is preloaded by a thin-walled phosphor-bronze cylindrical spring and a brass round nut, and is placed in contact with the ring-shaped piezoelement and the brass support base. The brass base, the bronze thin-walled cylindrical spring, and the brass round nut are electrically insulated using a nylon insulator. The driving voltage of the piezoactuator is applied between the brass round nut (+) and the brass base (G).

When designing, the purpose of the nylon insulator was electrical insulation, but it was found that it had another important function. When I used an aluminum insulator coated with insulating alumite to test the insulating materials to replace the nylon insulator, the modulation became unstable although there was no problem in the insulating property (substantial temporal change in amplitude occurred). The cause was the vibration of the mirror holder transferred to the phosphor-bronze thin-walled cylindrical spring and to the brass round nut through strong nonlinear screw contact. This resulted in complex resonance. The soft nylon insulator functioned as a damper that greatly reduces this resonance.

### 5.3 Anti-vibration mechanism

As mentioned earlier, to achieve a stable function of the iodine stabilized He-Ne laser, the laser resonator length change caused by vibration and acoustics of the external environment must be kept under the diameter of an atom (0.3 nm) or less. As an assumption for development of the



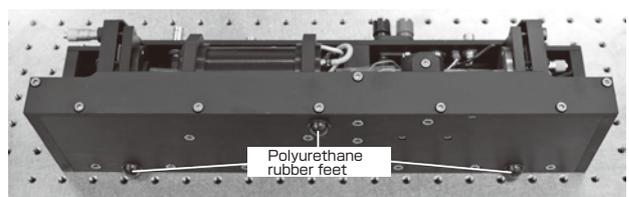
**Fig. 13 Modulation actuator (after improvement, structure with low harmonic distortion).**

vibration mechanism, it is necessary to establish a method for detecting and evaluation the slight changes of the resonator length. Following is a brief explanation of the slight change in resonator length and the anti-vibration mechanism.

The optical intensity change of laser output is measured for the stabilization of the laser wavelength, and this is amplified and connected to loudspeakers. The intensity change caused by wavelength modulation (6 MHz converted to optical frequency) of frequency  $f$  (3 kHz) can be heard as sound. The change of wavelength by wavelength modulation is converted to intensity change at laser wavelength – output curve  $I(\lambda)$  and is heard as sound. At the same time, the resonator length change caused by external vibration also causes wavelength change, and that also can be heard. The amplitude of sound is proportional to the wavelength change or change in the resonator length. Therefore, if the sound of the external vibration can be kept to 1/10 of the amplitude of sound caused by wavelength modulation (1/100 in intensity, -20 dB), stable operation of the iodine stabilized He-Ne laser can be achieved. The metal housing of the iodine stabilized He-Ne laser was placed directly on the optical table with a honeycomb structure (the surface is 5 mm thick stainless plate). The sound from the loudspeaker when the table surface is scratched with a coin or tapped lightly was similar to the sound heard when a person pressed an ear against the table surface.

To assess the performance of anti-vibration rubber, aluminum plate with thickness of 1 cm (same as the bottom plate of the laser body) was placed on the table, and the sound heard when the table surface was scratched or tapped while pressing the ear against the plate was checked. Next, the anti-vibration rubber to be assessed was placed beneath the metal plate, and the degree of reduction of sound was checked.

Three hemisphere rubber feet with radius of 7 mm made of polyurethane, which showed excellent sound insulation in the above simple experiment, were placed at the bottom of the housing of the new iodine stabilized He-Ne laser (Fig. 14). When it was placed directly on the table and operations such as optical axis adjustment were done, the wavelength stabilization became unlocked due to vibration, but with the anti-vibration effect of the rubber feet, there was hardly any effect on wavelength stabilization due to ordinary operations on the table.



**Fig. 14 Sub-nanometer vibration-proof mechanism (rubber feet).**

## 6 Performance of the developed national standard and its utilization

The new iodine stabilized He-Ne laser is used as the national standard of Japan at AIST and as the standard of the calibration service providers that provide the wavelength standard. This laser has new, original mechanisms as explained above. Its greatest characteristic is that it is supplied as a do-it-yourself assembly kit rather than a whole product. As mentioned earlier, what is important for a standard using the quantum mechanical property of iodine molecule absorption line is the technology of extracting the standard (wavelength) from that property. To put this thinking to practice, the author published *Monograph of Metrology*<sup>[6]</sup> that explains the details and plans of the new iodine stabilized He-Ne laser. Based on this monograph, one can create lasers that will satisfy the recommendations of CIPM. However, although the parts are generally available, it is hard work to procure all of the necessary parts to fabricate the laser. Therefore, we gathered all the necessary parts and distributed them for a charge to companies as a do-it-yourself kit. Since it is a kit, it is guaranteed that each member satisfies the specification, but the final performance as a standard (absolute wavelength and uncertainty) depends on the owner's technology. Technological instruction for assembly is provided by request. By distributing this kit, we realized a "bargain" price as a national standard at 2,000,000 yen a set including the laserhead and the controller (control part is provided in almost completed form).

The attempt to transfer the technology of wavelength standard is also done internationally. AIST has been working on the project to assist the establishment of the National Institute of Metrology Thailand (NIMT). The first kit of the iodine stabilized He-Ne laser was delivered to NIMT through this project. Current, similar technological transfer is planned for the national standard labs in the ASEAN countries.

This laser was used for international comparison, and was found to have excellent feature unseen in other lasers, as a standard that follows the recommendation of CIPM. One of the recommended parameter of CIPM states that the temperature of the wall of the tube of the iodine cell must be maintained at  $25 \pm 5$  °C. This temperature is almost the same as the environmental temperature, and the iodine stabilized He-Ne laser should function in the temperature range of  $25 \pm 5$  °C. The new laser fulfills this condition. It is capable of maintaining wavelength stability in the range of  $25 \pm 5$  °C, and the wavelength change showed good results where uncertainty stayed at half or less of the recommended by CIPM (5 kHz or less converted to optical frequency). On the other hand, many lasers including the conventional laser require strict stabilization of environmental temperature to fulfill the uncertainty recommended by CIPM. The change in tilt angle of the laser mirror due to the expansion

and contraction of the ring-form piezo stack actuator used in many lasers may become a factor of change in laser wavelength. To prevent the change in the tilt angle, it is necessary to conduct strict environmental temperature stabilization to minimize the expansion and contraction of the actuator, in addition to the use of ultra low expansion material for the laser main body. Once, we conducted the international comparison by gathering the iodine stabilized He-Ne lasers of various nations that were used as national standards in one place. During that session, the temperature of the room where the lasers were set up changed about 2 °C due to the malfunction of the air conditioner. Many lasers became unlocked and stopped functioning while the new laser continued to perform without unlocking, and demonstrated its high performance.

## 7 Conclusion

The author became involved with iodine stabilized He-Ne laser as a user. In "developing the precision interferometer," my initial topic of research, my latent desire was to improve the usability and reliability of the iodine stabilized He-Ne laser that was the standard light source for wavelength. When I saw the portable laser from New Zealand, the desire became manifest and I started the research as explained earlier. However, if I was not a user but a developer of the conventional laser, I am likely to have said, "This is the national standard and I don't care about the usability. Rather, it is more important to develop the next-generation standard with higher precision."

I think the greatest factor that made me set the direction of the research – to achieve special function by using the universal parts in unconventional, special ways – was the fact that the improvement of the iodine stabilized He-Ne laser was not my official topic. In the early phase of the research, I had extremely low budget and I had no time schedule or obligation to come up with a result. This allowed me to try bold experiments and that set the direction of research. If a research plan was written as an official project with formal budget request, I am certain I would have made safe choices of using special parts and special materials in a conventional manner.

I mentioned in the previous chapter that a "bargain" price was achieved by providing the iodine stabilized He-Ne laser as a do-it-yourself kit. However, the true significance of DIY is the increased motivation of the users. The assembly of the laser is done as a training session, where many participants work on it with enthusiasm and become totally absorbed in it. The author tells the participants who finish the session and are taking the assembled and adjusted iodine stabilized He-Ne laser home, "This laser is not a standard but just a thing. Standard is the technology you have learned and your will to maintain this standard."

## Notes

**Note 1)** Currently, the SI Base Units of length is the meter (metre). The metre is the length of the path travelled by light in vacuum during a time interval of  $1/299\,792\,458$  of a second.

**Note 2)** Currently, the helium-neon laser in which the emission frequency (wavelength) is stabilized to the specific absorption line of the iodine molecule is commonly used as the primary standard of length. The frequency recommended is  $\nu = 473,612,353,604$  kHz. Wavelength  $\lambda$  is calculated by dividing the speed of light  $c$  ( $299,792,458$  m/s) by  $\nu$ .  $\lambda = c/\nu$

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## Author

### Jun Ishikawa

Completed the master's course at the Interdisciplinary Graduate School of Science and Technology, Tokyo Institute of Technology in 1982. Joined the National Research Laboratory of Metrology, Agency of Industrial Science and Technology, Ministry of International Trade and Industry in 1983. Worked on the development of precision interferometer, and was exposed to the iodine stabilized He-Ne laser as a light source for wavelength standard. Joined the National Metrology Institute of Japan, AIST in 2001. Transferred to the Digital Manufacturing Research Center in 2006, where he worked on visualization of skill at the site of manufacturing. Currently, leader of research team for measurement and analysis technology. Received Ichimura Academic Award (The New Technology Development Foundation) in 2006. Received the Commendation for Science and Technology by the Minister of Education, Culture, Sports, Science and Technology in 2006.



## Discussion with Reviewers

### 1 Addition to research objective and scenario

#### Comment (Akira Ono, AIST)

For *Synthesiology*, please add the “objective of research” and “scenario for achieving the objective.”

#### Answer (Jun Ishikawa)

As you indicated, I added a new section for “objective of research” and “scenario for achieving the objective.” However, this research was started without a scenario (plan), and I frankly explained the course of events at the time.

### 2 Use of AIST Monograph of Metrology in the reference

#### Question and comment (Akira Ono)

I think Reference 6 is extremely important in terms of diffusion of this technology. While there is a tendency to write up only the highlights of the original technology in research papers and be done with it, I don't think I've seen a complete disclosure of necessary information to enable reproductive fabrication of the device. What was your motivation for writing Reference 6, and how is it being utilized?

#### Answer (Jun Ishikawa)

Unlike the International Prototype Metre Standard Bar, which is an artificial object, the essence of the laser wavelength standard that utilizes the quantum mechanical property of the iodine molecule is in the technology of extracting the optical wavelength from the quantum mechanical property. Therefore, the maintenance and transfer of the standard is the maintenance and transfer of the technology. The *AIST Monograph of Metrology* was written for the purpose of maintenance and transfer of the technology for the iodine stabilized He-Ne laser. The paper was revised to reflect this thinking.

### 3 Mass and size of the standard

#### Question (Akira Ono)

You mention that the national standard can be taken aboard an airplane for transfer. That must be extremely convenient for conducting international comparisons smoothly. What is the mass and size of the newly developed laser? Can it be taken aboard the airplane cabin?

#### Answer (Jun Ishikawa)

The body of the laser is 420 mm in length, 105 mm in width, 95 mm in height, and 5.3 kg in weight. The control device is 400 mm in depth, 420 mm in width, 100 mm in height, and 7.5 kg in weight.

When boarding the airplane, the laser body is taken aboard the cabin as a hand-carry item, and the control device is checked in. If we concentrate on downsizing and weight reduction, a smaller and lighter laser can be made, but I opted for this size due to the ease of fabrication, assembly, and adjustment.

At this point, the greatest barrier to hand-carrying the device on the airplane is the regulation after 9.11. Since the wavelength-stabilized laser is a special device which people are not familiar with, I have a very hard time explaining what it is.

### 4 Uncertainty of laser wavelength

#### Question (Akira Ono)

What is your assessment of the uncertainty of the laser wavelength of the new laser? I imagine that it is smaller than the value of uncertainty recommended by CIPM. If possible, can you show the budget table along with the factors of uncertainty?

#### Answer (Jun Ishikawa)

Table “a” shows the budget table of the uncertainty of iodine stabilized He-Ne laser recommended by CIPM. Of the uncertainties shown in the table, I think the estimate of the uncertainty caused by one-directional optical intensity within

the resonator is too large. However, I think this includes the gas lens effect and gas prism effect that were difficult to assess separately in the conventional iodine stabilized He-Ne laser that had problems with linearity in its control mechanism. The researcher who has been working on iodine stabilized He-Ne laser for a long time at the Bureau International des Poids et Mesures acknowledged this point.

Since the linearity of the control mechanism of the new laser is excellent, the gas lens and gas prism effects can be separated, and the uncertainty budget will be as shown in Table “b”. To reduce the uncertainty due to gas lens and gas prism effects, it is necessary to precisely fabricate the geometric shape of the laser discharge tube, and such laser tube is not available in reality. The effect of purity of iodine was reduced by improving the filling process of the iodine cell, and the uncertainty of beat frequency measurement was reduced by improving the measurement technology.

### 5 Precision of the standard owned by the calibration service provider

#### Question (Akira Ono)

You say that several calibration service providers in Japan own the iodine stabilized He-Ne laser as their own standards, and I believe they are brought to AIST for regular calibration. When the calibration was conducted, what was the difference compared to the wavelength (frequency) of the national standard at AIST. Did it fall within the range of uncertainty assessed for the new laser, or did some of them surpass the range?

#### Answer (Jun Ishikawa)

The new lasers were all assembled and adjusted under the author’s technological instruction at AIST. Therefore, the frequency difference of 5 kHz or less against the national standard was checked at the time of shipment. Except for the case where the emission ceased due to clouding of the optical window, I confirmed that the initial performance was almost completely maintained at the time of recalibration.

**Table a Uncertainty of the iodine stabilized He-Ne laser recommended by the CIPM.**

Parameter	Recommended value	Tolerance	Coefficient	Uncertainty(kHz)
Iodine cell				
Wall temperature	25 °C	5 °C	0.5 kHz/°C	2.5
Cold-finger temperature	15 °C	0.2°C	-15 kHz/°C	3.0
Effect of iodine purity				5.0
Frequency modulation width peak-to-peak	6 MHz	0.3 MHz	-10 kHz/MHz	3.0
One-way intracavity beam power	10 mW	5 mW	<1.0 kHz/mW	5.0
Uncertainty of beat frequency measurement				5.0
Combined standard uncertainty				10.0

**Table b Uncertainty of the new iodine stabilized He-Ne laser.**

Parameter	Recommended value	Tolerance	Coefficient	Uncertainty(kHz)
Iodine cell				
Wall temperature	23 °C	2 °C	0.5 kHz/°C	1.0
Cold-finger temperature	15 °C	0.1 °C	-15 kHz/°C	1.5
Effect of iodine purity				2.0
Frequency modulation width peak-to-peak	6 MHz	0.2 MHz	-10 kHz/MHz	2.0
One-way intracavity beam power	10 mW	2 mW	<1.0 kHz/mW	2.0
Uncertainty of beat frequency measurement				0.0
Gas lens effect, gas prism effect				5.0
Combined standard uncertainty				6.3

### 6 What happened to the overseas researcher

#### Question (Akira Ono)

The researcher from New Zealand inspired you to carry out your idea. What kind of research did this researcher do afterwards? Can you answer to the extent you know?

#### Answer (Jun Ishikawa)

After that, due to the administrative reform in New Zealand, many research institutes were reduced, abolished, or merged, and I’ve heard from his successor that the researcher transferred to a private company. New Zealand’s iodine stabilized He-Ne laser is currently an American product, and it’s a little sad to hear that the laser that he used for the international comparison is no longer used and sits in the corner of the lab.

### 7 Examples of Product Realization Research conducted by overseas national standards laboratory

#### Question and comment (Akira Ono)

The universalization research for the national standard that you conducted can be considered a *Product Realization Research*. I don’t think there are so many examples in the AIST Metrology Standard Groups engaging in research with a clear goal of product realization. However, some researches such as “remote calibration” are being done in the mid-tier of the traceability system involving the calibration service providers, and it seems that you are strongly aware of the importance of *Product Realization Research*.

Around the world, to the best of the author’s knowledge, is there any example, other than New Zealand, of such *Product Realization Research* done by a national standard laboratory?

#### Answer (Jun Ishikawa)

Attempts at product realization of iodine stabilized He-Ne laser was done from its early stages (around 1980), but it mostly involved the manufacturers almost entirely copying the R&D product from the laboratory to create the product. As a result, there were problems in price, size, and operability, and it was not of high quality as a product. The laser from New Zealand was developed from the user’s standpoint for the purpose of using it for international comparison. It had outstanding portability and operability at that time. Although the laser never became a product in New Zealand, I think it is essential to carry out the universalization research from the user’s standpoint to achieve successful product realization. I don’t know so many examples where universalization research was carried out from the user’s standpoint for a device with highly specialized purpose such as the iodine stabilized He-Ne laser. However, product realizations for more general devices (such as the mirror holder) have been done at national standards laboratories, and these have superior performances compared to the conventional products from manufacturers.

# How the reliable environmental noise measurement is ensured

## — Development of acoustic standards and a new calibration service system —

Ryuzo Horiuchi

[Translation from *Synthesiology*, Vol.2, No.4, p.288-298 (2009)]

To ensure the reliable results in acoustic measurement such as environmental noise measurement, NMIJ/AIST has developed essential calibration techniques and established a traceability system based on the Japanese Measurement Law with acoustic standards at the highest level of accuracy. A new calibration service for reliable acoustic measurements under this system realized a minimum uncertainty in the environmental noise measurements, indispensable to sustain high quality of our daily life.

**Keywords :** Environmental noise measurement, laboratory standard microphone, pressure sensitivity, coupler reciprocity method, measurement microphone, sound level meter, free-field sensitivity, anechoic chamber

### 1 Introduction

In some way or another, we are exposed to sound in our daily life. Sound disturbing our conversation or uncomfortable sound is regarded as noise. Noise affects our daily life in many ways such as sleep disturbance or inefficiency in working and in some cases will result in serious health damage of hearing loss.

The Japanese Basic Environment Law provides basic principles of environmental preservation and the measures for its realization to secure our healthy and cultural life, and to contribute to the welfare of human beings. The law deals with environmental problems such as noise pollution and air pollution, and gives regulations and standards on the environmental noise. The Noise Regulation Law provides regulations for the noise from specific plants, construction machines and cars. Environmental quality standards to be achieved by the government are set for traffic, Shinkansen (Bullet train) and airplane noise. NMIJ/AIST has been requested to solve technical problems necessary for the reliable environmental noise measurement and thus to sustain high quality in our daily life from the standpoint of environmental noise.

Physically speaking, sound wave is a phenomenon in which the vibration of a sound source causes the vibration of a medium (air) surrounding it and the vibration of the medium is spatially transmitted<sup>[1]</sup>. The vibrational transmission causes change in time and spatial distribution of medium density, resulting in pressure fluctuation. Sound pressure is defined as pressure fluctuation from static pressure caused by the sound wave. Sound pressure is a main physical quantity in acoustics and thus its precise measurement is essential<sup>[2]</sup>.

In many cases, the magnitude of the sound pressure is expressed as sound pressure level<sup>Term 1</sup>. Decibel and dB is used as its unit and symbol, respectively. If two types of test sounds have the same sound pressure level but different frequency (pitch), they are recognized as different loudness because our hearing is dependent on frequency. Frequency weighting which imitates our hearing is named frequency weighting A and the sound pressure level considering frequency weighting A is named A-weighted sound pressure level, or more generally, the sound level. The unit of the sound level is decibel, the same as the sound pressure level. (In the past, the unit of the sound level was phon but it is internationally standardized to decibel now.) The sound level is used to evaluate the environmental noise or the noise from instruments<sup>[3]</sup>.

As shown in Fig. 1, the sound pressure level or the sound level is measured by acoustic measuring instruments such as measurement microphones<sup>[4]</sup> or the sound level meters<sup>[5,6]</sup>. The sound pressure can be got from the output voltage of the measurement microphone which is used as a sensor of the sound pressure. The sound level meter can directly display sound pressure levels or sound levels to be measured because it works as a sensor of the sound pressure and calculates those quantities.

Typical end-users of sound level meters are as follows; (1) providers who officially verify the environmental measurement results by a certificate of the measured sound level or the environmental measurement specialists who constitute the providers, (2) local autonomies who measure the environmental noise in various districts, (3) manufacturers who measure acoustic characteristics of their products, (4) scientists in universities or institutes who conduct acoustic measurements.

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The Japanese Measurement Law has regulated “specific measuring instruments” essential for business dealings or certification. To realize the reliable measurement, the law provides items to be tested for structure and measurement accuracy, and the corresponding criteria to be reached. The sound level meter is designated as one of the specific measuring instruments and has been tested. At present, about fifty thousand sound level meters have passed the testing and they are used for official noise measurement.

In the testing procedure of the sound level meter, the sound pressure applied to the microphone, namely the sound pressure at the tip of the sound level meter is precisely measured to evaluate the indication of the sound level. A laboratory standard microphone is used to determine this sound pressure. As shown in Fig. 2, it is a special measurement microphone superior to the others in stability<sup>[7,8]</sup>. In other words, sensitivity of the laboratory standard microphone as a sensor of the sound pressure, namely the ratio of the output voltage to the input sound pressure, is adopted as the national standard in acoustic measurement (the acoustic standard). The laboratory standard microphone used as a reference to test the sound level meter is named the reference standard for the sound level. NMIJ/AIST has calibrated a lot of reference standards for the sound level for nearly half a century including the time of the former Agency of Industrial Science and Technology / Electrotechnical Laboratory<sup>[9,10]</sup>.

The Japanese testing system has allowed only sound level meters which passed the testing to be used for business dealings and certification, and this will be continued into the future. However, the testing does not cover the concept of uncertainty which has been internationally expressed in recent years. Uncertainty of calibration results is not expressed in the testing reports of the reference standards for the sound level. In the testing procedure of the sound

level meter as a specific measuring instrument, it is judged to fulfill the criterion only by comparing its indication of the sound level with the value determined by the reference standard for the sound level and determining that it is within the normal tolerance.

Uncertainty is now introduced into IEC and ISO international standards and JIS, which prescribe specification and calibration methods of the measuring instruments. From the scientific point of view, any measurement result essentially has some extent of uncertainty. Uncertainty of the measurement results should be properly stated to ensure objective reliance. Moreover, World Trade Organization / Technical Barriers to Trade (WTO/TBT) arrangement was made among the countries including Japan in 1995. Each country took similar responsibility to ensure the conformity to the international standards of measuring instruments in its country. Each country was required to develop the domestic traceability system<sup>Term 2</sup> of measuring instruments to the national standard and to verify technical equivalence of national standards among the countries.

Similarly in the acoustic area, uncertainty evaluation of acoustic measuring instruments became essential to verify their conformity to the corresponding standards<sup>[11]</sup>. Establishment of a measurement traceability system (Fig. 3) was indispensable to verify that end-users’ acoustic measuring instruments fulfill the standards.

In addition to the environmental noise measurement, measurement of the noise from household appliances (refrigerator, washing machine, vacuum cleaner, etc.) and information equipment (copying machine, personal computer, printer, etc.) has recently become important. Measurement microphones are used to evaluate the noise from these kinds of equipment<sup>[12,13]</sup>.

Manufacturers of household appliances and information equipment cannot obtain reliable data on the acoustic



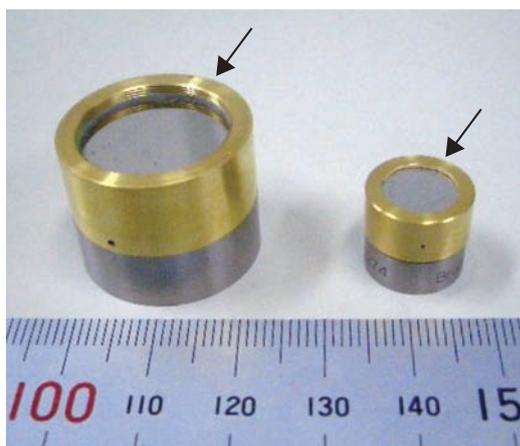
**Fig. 1 Measurement microphone (left) and sound level meter (right).**

Measurement microphone is a sensor of sound pressure and generates the output voltage proportional to the sound pressure applied to the diaphragm (circular surface in the left figure). Type WS1 and WS2 microphones are available for the audible frequency range (20 Hz to 20 kHz). They are different in size and in suitable frequency range. The larger (left) is type WS1. Sound level meter senses sound pressure by the measurement microphone at the tip (left in the right figure) and calculates sound pressure level or sound level. Sound level meter consists of microphone, amplifier, frequency weighting circuit, calculation circuit and indicator.

characteristics of their own products and ensure the quality of the products internationally until their acoustic measuring instruments are verified to be in conformity with the international standards or the equivalent JIS. The establishment of a traceability system for acoustic measuring instruments was an essential technology to develop the products envisioned on a road map of future technical development by the industrial world, namely products with high quality which are environmentally friendly and safe. Such a change of needs in the industrial world resulted in the establishment of a new calibration service system of acoustic standards, which is different from the traditional testing system.

For the reliable environmental noise measurement, end-users should not only use the traceable measuring instruments but evaluate the influence specific to the measurement site such as environmental conditions (temperature, static pressure and wind) and indirect sound from the surroundings. Uncertainty related to a sound field (a space in which the sound wave is transmitted) containing indirect sound has not been technically evaluated up to this point. Influence of indirect sound depends on relative positions between acoustic measuring instruments and sound reflecting objects such as the ground or buildings. In the past, measured data were averaged by changing the position of the measuring instrument. Uncertainty caused by indirect sound could be decreased by this method, but the obtained data were not reliable enough because the remaining uncertainty could not be quantitatively evaluated.

The calibration of acoustic measuring instruments had the same technical problem. Acoustic measuring instruments are usually calibrated within an anechoic chamber which



**Fig. 2 Laboratory standard microphone.**

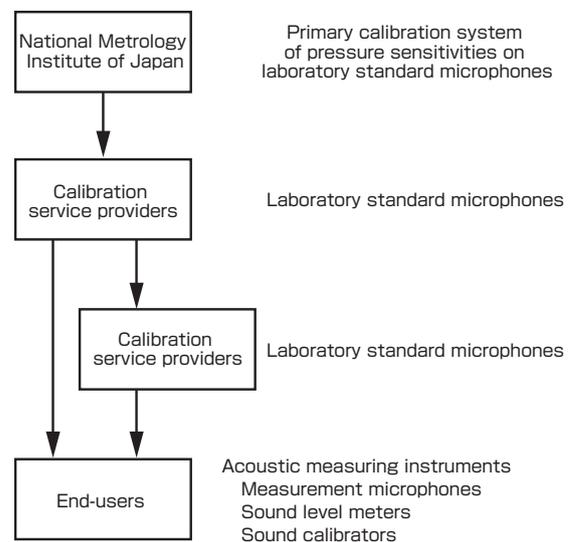
Laboratory standard microphone is one of the measurement microphones but its sensitivity must be sufficiently stable compared with the others because it is required to be reliable as an acoustic standard. It has a special structure around the diaphragm (gold part and indicated by an arrow in the figure) to protect the diaphragm when it is fitted to the coupler for the sensitivity calibration as will be explained later. Left is type LS1P and right is LS2aP microphone, respectively.

is designed to minimize the influence of indirect sound. However, even a high-performance anechoic chamber cannot realize a space completely free from indirect sound. Indirect sound was the main cause of uncertainty in the calibration of acoustic measuring instruments. JIS of sound level meters<sup>[6]</sup> requires uncertainty decrease by placing the sound source and the sound level meter at several positions and by averaging the measured data. However, it is just one of the procedures to judge if the specification of the sound level meter fulfills the criterion, and the uncertainty still remained not properly evaluated.

Considering such a situation, NMIJ/AIST developed the technique necessary to evaluate the uncertainty of the sound field caused by indirect sound and thus solved the technical problem.

Furthermore, there was one more technical problem in the calibration of acoustic measuring instruments. Acoustic standards realized so far did not have measurement uncertainty small enough to evaluate the conformity of some acoustic measuring instruments to the corresponding standard. Development of advanced (high-precision) acoustic standards was essential to decrease the uncertainty.

In the following chapters, research results are described which were done to solve these problems and to realize the reliability of environmental noise measurement.



**Fig. 3 Schematic of traceability system in acoustic measurement.**

Laboratory standard microphones possessed by a high-ranking calibration service provider are calibrated by using a primary calibrated system NMIJ developed. Lower-ranking provider's microphones are calibrated in comparison with higher-ranking provider's microphones. End-users' acoustic measuring instruments are calibrated in comparison with higher or lower ranking provider's microphones.

## 2 Elemental technologies and research scenario

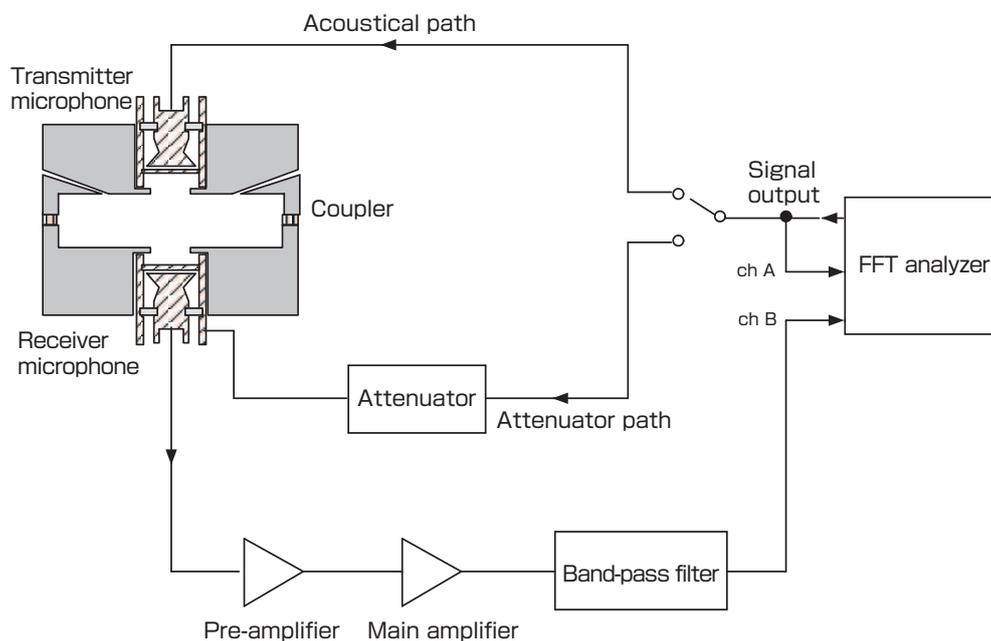
Elemental technologies are necessary to solve the two technical problems mentioned in the last chapter and to achieve a research goal of realizing the reliable environmental noise measurement.

The first elemental technology is the uncertainty decrease in sensitivity calibration of laboratory standard microphones. Calibration uncertainty of laboratory standard microphones realized by NMIJ/AIST thus far was around 0.1 dB at the smallest. According to JIS, however, calibration uncertainty of a high-grade sound calibrator used to check sound level meters<sup>[14]</sup> (a portable sound source which generates the given sound pressure to calibrate the instruments) was less than 0.1 dB. The laboratory standard microphone with an uncertainty of 0.1 dB was not suitable as a reference to evaluate the performance of the sound calibrator. Therefore, an advanced calibration system of laboratory standard microphones became essential. Signal-to-noise ratio of the calibration system was improved by introducing the digital signal processing technique etc. and the uncertainty was decreased to 0.04 dB.

The second elemental technology is the development of the method necessary to evaluate the uncertainty caused by the imperfection (existence of indirect sound) of the sound field which is used to calibrate acoustic measuring instruments. The developed method made it possible to evaluate the uncertainty quantitatively by visualizing the influence of the indirect sound and to remove unnecessary indirect sound by using the digital signal processing technique.

Lastly, the traceability system of acoustic measuring instruments was required to return these research results to society. It is not until the traceability system is established that the advanced calibration technique of laboratory standard microphones and acoustic measuring instruments gives more reliable measurement results to end-users. NMIJ/AIST established the traceability system, providing acoustic standards as the highest level of accuracy, and calibration service providers could calibrate end-users' acoustic measuring instruments using these acoustic standards.

The traceability system is required to confirm the measurement capability of the constituent organizations in each level, namely NMIJ/AIST and calibration service providers. NMIJ/AIST internationally participated in several round robin tests among the national metrology institutes and



**Fig. 4 Schematic of primary calibration system of laboratory standard microphones by the coupler reciprocity method.** Coupler reciprocity method uses two laboratory standard microphones, one as a transmitter and the other as a receiver. Sound wave is generated from the diaphragm of the transmitter by applying the input voltage, arrives at the diaphragm of the receiver through a cavity of the coupler and the output voltage is detected at the receiver. Sensitivity product of the two microphones are obtained from the cavity volume of the coupler and the voltage ratio between the input terminal of the transmitter and the output terminal of the receiver, by using a principle of an electro-acoustic transducer that the sensitivity of the transmitter is equal to that of the receiver. Introduction of one more laboratory standard microphone enables the sensitivity of each microphone to be determined by measuring the voltage ratios for the three combinations of the transmitter and the receiver. This method requires cancellation of influence caused by the output impedance of the receiver and the gain of the amplifier to measure the open-circuit output voltage of the receiver precisely. Thus, the calibration system has two signal paths, namely the acoustical path and the attenuator path. Ratio of the receiver's output voltages between the two paths cancels this influence.

also technically supported the tests for domestic calibration service providers. Constituent organizations were assessed for their calibration procedures and uncertainty evaluation methods by a third party to verify their measurement capability objectively.

Development of individual elemental technologies and their integration by the traceability system resulted in an international scheme of providing the reliable environmental noise measurement results for end-users.

### 3 Microphone as national standard

#### 3.1 Primary calibration of pressure sensitivity

The microphone as a national standard (laboratory standard microphone) has two requisites: the stability of pressure sensitivity<sup>Term 3</sup> and the established method for precise absolute calibration (primary calibration) of the pressure sensitivity. The coupler reciprocity method<sup>[15]</sup> is used for primary calibration of pressure sensitivities on laboratory standard microphones. As shown in Fig. 4, the sound wave is kept within the acoustic coupler (a cavity with small volume) during calibration. Other calibration methods were not adopted because one cannot cover the whole audible frequency range (20 Hz to 20 kHz)<sup>[16]</sup> and the others have a drawback of having larger uncertainties<sup>[14,17]</sup>.

NMIJ/AIST introduced digital signal processing technique into the coupler reciprocity method and improved the calibration uncertainty from the point of view of noise reduction, attenuator calibration and cross-talk minimization<sup>[18]</sup>.

Firstly, digital signal processing by using an FFT analyzer<sup>Term 4</sup> was adopted as a new noise reduction technique because analogue signal processing by a traditional filter with narrow bandwidth had a problem in measurement repeatability. Synchronous average method by using a built-in signal source of the FFT analyzer improved measurement repeatability from 0.02 dB to 0.007 dB in the main frequency range and reduced measurement time by half.

Secondly, small output voltage (0.1 mV to 0.8 mV) which was attenuated to adjust a signal level could not be directly measured with small uncertainty to that point<sup>[19]</sup>. If the ratio of attenuation is pre-determined, small uncertainty can be ensured with only the measurement of large input voltage applied to the attenuator. Thus, the advanced calibration method for attenuators was developed by using the FFT analyzer and it decreased the uncertainty from 0.01 dB to 0.001 dB.

Lastly, measurement circuits were re-designed to minimize the cross-talk. It is one of the uncertainty factors, meaning that a signal is wrongly mixed due to bypassing the unexpected paths. Severe measures were taken as in the high

frequency circuits and the uncertainty caused by the cross-talk was decreased from 0.01 dB to 0.001 dB.

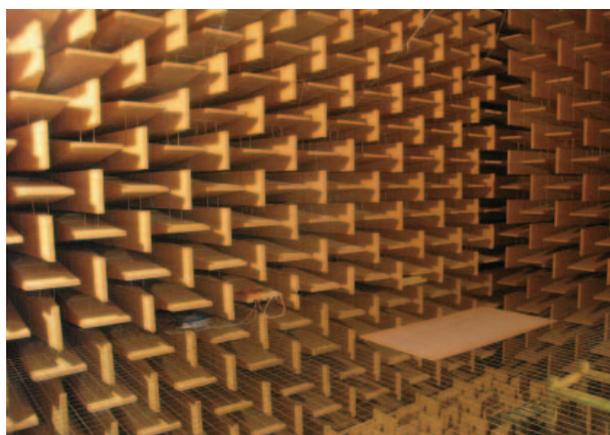
#### 3.2 Instability of pressure sensitivity

However, deviation of the pressure sensitivity still remained after the calibration system was improved. The author supposed that the cause of instability might be inherent in the microphone to be calibrated and considered various causes to verify this hypothesis. Theoretical analysis on frequency characteristics of the sensitivity deviation revealed that the microphone is deformed when contacting surfaces between the microphone and the coupler are sealed with grease and that the change of microphone's acoustic characteristics results in instability of the pressure sensitivity<sup>[20]</sup>.

Such deviations were remarkable to a specific type of domestic laboratory standard microphones and thus this type was not adopted as an acoustic standard<sup>[21]</sup>. At present, the uncertainty due to instability of the microphone sensitivity is 0.012 dB for LS1P microphones and 0.008 dB for LS2aP, respectively (Refer to Fig. 1 for the difference between LS1P and LS2aP microphones).

#### 3.3 Measurement uncertainty of pressure sensitivity

As a result of improvement, of the uncertainty components reformable at the state of the art, the influence could be minimized to a negligibly small level. Remaining uncertainty components are the instability of the microphone sensitivity and the internal volume of the coupler. Volume uncertainty of the coupler used for LS1P microphones (its internal volume is approximately 20 cm<sup>3</sup>) is 0.008 dB and that for LS2aP (1 cm<sup>3</sup>) is 0.015 dB, respectively. In the main frequency range, the uncertainty (95 % level of confidence) of the pressure sensitivity for both LS1P and LS2aP microphones is evaluated to be 0.04 dB. This uncertainty is half of what it was before<sup>[22]</sup>.



**Fig. 5 Anechoic chamber of NMIJ/AIST.**

Many sound absorbing wedges made of glass wool protrude from the inside wall of the anechoic chamber. Plywood was put on the wire meshed floor for a better view.

## 4 Evaluation of sound field

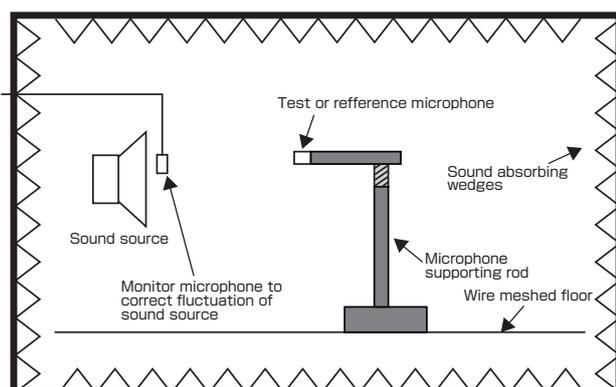
### 4.1 Indirect sound

Secondary calibration of acoustic measuring instruments is made in comparison with a reference standard microphone, usually within an anechoic chamber. A lot of sound absorbing wedges protrude from the inside wall of the anechoic chamber including its ceiling and bottom to minimize indirect sound. A floor necessary to carry measuring instruments into the anechoic chamber has a special structure of a wire meshed floor<sup>Term 5</sup> to decrease sound reflection<sup>[23]</sup>. However, actual sound field within the anechoic chamber is still influenced by indirect sound, due to incompleteness of sound absorbing wedges and the wire meshed floor. For the NMIJ/AIST's facility shown in Fig. 5, the degree of indirect sound is approximately 1 to 2 % of the direct sound. Furthermore, structures necessary to fix the reference microphone (laboratory standard microphone with pre-determined free-field sensitivity<sup>Term 6</sup>) or acoustic measuring instruments to be calibrated also influence sound reflection. Deviation of the sound field from an ideal situation due to indirect sound cannot be theoretically estimated and thus the corresponding uncertainty must be experimentally evaluated.

### 4.2 Secondary calibration of acoustic measuring instruments

Description in the following sections of chapter 4 is focused on calibration of measurement microphones. However, a similar approach can be applied to sound level meters.

Two secondary calibration methods are applicable to acoustic measuring instruments, namely sequential method and simultaneous method<sup>[24]</sup>. Both methods have the common



**Fig. 6 Schematic of secondary calibration system of measurement microphones by the sequential method.**

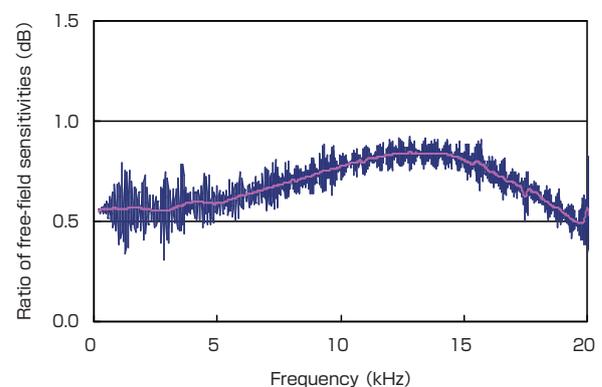
A lot of sound absorbing wedges protrude from the inside wall of the anechoic chamber to minimize indirect sound. However, it is quite difficult to realize an ideal free-field even in the high-performance anechoic chamber. As will be explained later, reflection from the object closest to the test or reference microphone has dominant influence. In this measurement system, reflection is mainly caused by the upper end of vertical rod which supports the microphone (area with oblique lines in the figure).

procedures as follows. The reference microphone and the test microphone (the measurement microphone to be calibrated) are placed ahead of a loudspeaker. Ratio of the output voltages between the two microphones, namely the ratio of sensitivities is measured. Sensitivity of the test microphone is determined as the product of this ratio by the sensitivity of the reference microphone. Two methods are different in the placement of the microphones.

In the sequential method, the reference microphone is replaced by the test microphone and the output voltage of the microphone is sequentially measured. This method assumes that equal sound pressure is applied to both microphones during the measurement. However, actual sound pressure fluctuates because the characteristics of the loudspeaker changes with the generation of heat and this phenomenon results in calibration uncertainty.

In the simultaneous method, both microphones are placed at close positions and are exposed to the sound field simultaneously. Fluctuation of sound pressure caused by the loudspeaker's instability does not cause a problem because the output voltage ratio of the microphones becomes stable due to the cancellation effect. Measurement time can be decreased by half compared with the sequential method. As described later, however, uncertainty related to the sound field increases because the two microphones are placed at different positions within the sound field; partly because sound pressure has a spatial distribution in the sound field and partly because the existence of one microphone disturbs the sound field to which the other microphone is exposed.

A drawback of the sequential method can be solved by placing a third microphone in front of the loudspeaker to monitor the fluctuation of sound pressure and by correcting the change. In this research, the sequential method was adopted because it can evaluate the uncertainty more



**Fig. 7 Frequency characteristics of ratio of free-field sensitivities between reference and test microphones.**

Influence of indirect sound is visualized as vertical fluctuation in the blue curve. The pink curve has smooth frequency characteristics because this influence was minimized by using digital signal processing technique as will be explained later.

precisely by introducing the monitor microphone. Figure 6 shows a schematic of secondary calibration system of measurement microphones by the sequential method.

#### **4.3 Visualization of influence by indirect sound**

Indirect sound reaches the microphone through a path different from direct sound and they interfere with each other. Thus on the frequency domain, sound pressure at the microphone position has local minimums and maximums alternately. Indirect sound adds small waves to the frequency characteristics of the microphone output voltage which would be inherently smooth without indirect sound. Influence of indirect sound changes with relative positions of the loudspeaker, microphone and sound reflecting objects. Reflection from the object closest to the microphone has dominant influence.

Influence of indirect sound also depends on the shape of microphone housing and even on the slight difference of positions between the reference and test microphones. Therefore, measured sensitivity ratio between the microphones has frequency characteristics as shown in the blue curve of Fig. 7. The amplitude of the waves is equal to the uncertainty caused by indirect sound and frequency of the waves corresponds to the distance between the sound reflecting object and the microphone. Frequency dependence of sensitivity ratio enabled the specification of the most influential object and taking the measures necessary to decrease indirect sound.

In some cases, influence of indirect sound was decreased by averaging the sensitivity ratio in the vicinity of the measurement frequency. This method is not appropriate from the point of view of calibration results and uncertainty.

#### **4.4 Reduction of influence by indirect sound – application of sound absorbing material –**

The author tried to decrease the influence of indirect sound simply by covering the sound reflecting object with sound absorbing material. This method was effective if the reference and test microphones belonged to the same type but it was not sufficient for the different types.

#### **4.5 Reduction of influence by indirect sound – application of digital signal processing technique –**

Uncertainty caused by indirect sound can be decreased if the indirect sound is separated and removed from the direct sound on the time domain, since the indirect sound reaches the microphone later than the direct sound. However, simple application of pulse waveform with short duration as an input signal cannot give sufficient signal-to-noise ratio because the energy of the waveform is essentially distributed to the frequencies other than the measurement frequency. In this research, NMIJ/AIST developed a virtual pulse method to solve this problem<sup>[25]</sup>. This method makes use of computer

simulations to determine time response which would be obtained by the application of pulse waveform. Sufficient signal-to-noise ratio can be ensured because a continuous waveform is used to measure the data necessary for simulation. The virtual pulse method could not be realized until digital signal processing by the FFT analyzer was introduced into the measurement.

In the method, time response is calculated on condition that a virtual pulse signal is applied to the system with transfer function as shown in the blue curve of Fig. 7. Only direct sound is taken from the calculated pulse response waveform by applying the time window function and it is transformed into the frequency domain. As a result, smooth frequency characteristics not influenced by indirect sound can be achieved as shown in the pink curve of Fig. 7.

After indirect sound was removed by digital signal processing technique, still remaining small uncertainty related to the signal processing, namely uncertainty caused by slight difference of parameters used in the signal processing were evaluated. These were such differences as the frequency bandwidth of the pulse waveform and duration and center position of the time window function to remove indirect sound.

### **5 Establishment of traceability system on acoustic measuring instruments by JCSS**

As described in chapter 1, a new calibration service system of acoustic standards, different from the traditional measurement management system based on the testing of sound level meters, was required. Basis of the new system was JCSS (Japan Calibration Service System) in the Japanese Measurement Law<sup>[26]</sup>. In the system, firstly NMIJ/AIST evaluates calibration uncertainty of laboratory standard microphones as national standards and calibrates laboratory standard microphones of calibration service providers in comparison with one of the national standards. Then calibration service providers calibrate end-users' acoustic measuring instruments in comparison with one of their laboratory standard microphones and ensure traceability to the national standards. Special attention was paid to the following points to establish the system.

- The system allows calibration service providers to develop individual measurement management procedures. In addition to the receiving supply of national standards directly from NMIJ/AIST, they can also get acoustic measuring instruments traceable to the national standards from other high-ranking calibration service providers. Furthermore, measurement management is possible by using acoustic measuring instruments other than laboratory standard microphones as working standards for daily calibration use.

- The system can introduce new technology fairly rapidly by reconsidering technical requirements essential for the assessment of calibration service providers whenever necessary.
- JCSS can verify the performance of acoustic measuring instruments within the audible frequency range (20 Hz to 20 kHz) while the testing of sound level meters was limited to a range of 20 Hz to 12.5 kHz.
- Calibration service providers are required to have technical performance suitable for essential players of the traceability system. Calibration service providers registered at present have years of accomplishments as a specific testing laboratory of sound level meters or specific manufacturers of them in the Japanese Measurement Law.

## 6 Validation of measurement capability

### 6.1 Validation of equivalence among national standards

In 1999, mutual recognition arrangement of measurement standards was made among countries including Japan<sup>[27]</sup>. In this arrangement, national metrology institutes of the countries approve the equivalence among national standards and accept calibration certificates of each other. Institutes of signatory nations are required to establish the quality system<sup>Term 7</sup> conformable to the international rule of ISO/IEC 17025<sup>[28]</sup> and to verify their measurement capability objectively by participation in international comparisons (round robin tests).

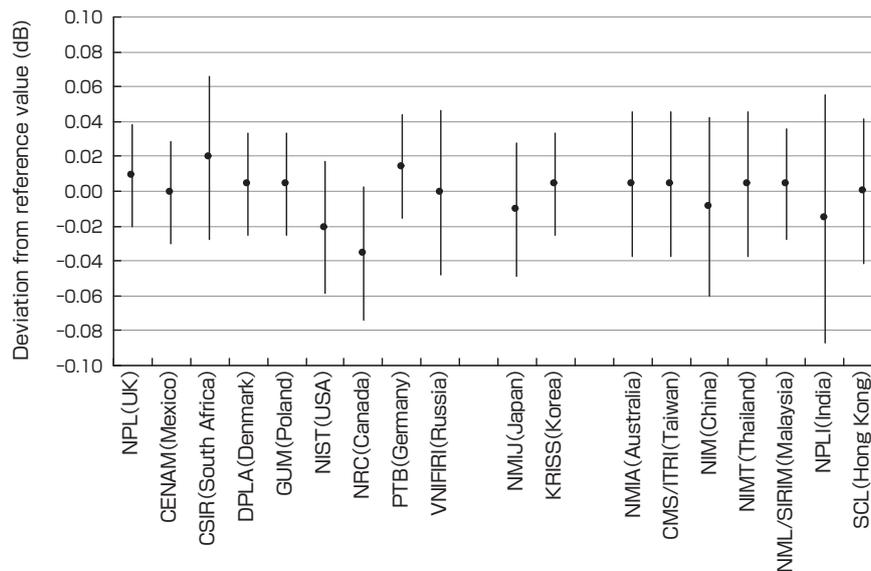
NMIJ/AIST established the quality system on self-developed acoustic standards. The National Institute of Technology and

Evaluation (NITE) assessed NMIJ/AIST from the point of view of conformity to ISO/IEC 17025, based on ASNITE-NMI (a program to accredit national metrology institutes). NMIJ/AIST was accredited in the field of acoustics in January 2003.

As to international comparisons, CIPM/CCAUV (International Committee for Weights and Measures / Consultative Committee for Acoustics, Ultrasound and Vibration) planned four international comparisons on laboratory standard microphones and NMIJ/AIST took part in all of them. International comparisons on pressure sensitivities of LS1P and LS2aP microphones have already finished and the results revealed that uncertainties (95 % level of confidence) declared by the main institutes including NMIJ/AIST range from 0.03 dB to 0.05 dB within the main frequency range<sup>[29,30]</sup>. Difference of the results between NMIJ/AIST and other institutes was less than the uncertainty evaluated by NMIJ/AIST. Thus, NMIJ/AIST could verify its equivalence to the other national standards.

The number of participants in the international comparisons organized by CCAUV is limited to around ten and all the national metrology institutes in the world cannot be included. Therefore, similar international comparisons were individually conducted in several areas of the world.

In Asia and Pacific area including Japan (this area is called APMP /Asia Pacific Metrology Programme), NMIJ/AIST acted as a pilot laboratory of the first international comparison for pressure sensitivity of LS1P microphones; NMIJ/AIST prepared the technical protocol, monitored



**Fig. 8 Results of international comparison at 1 kHz.**

Acronyms of national metrology institutes which participated in the international comparison are listed along the horizontal axis. Vertical axis shows deviation of each participant's results from the reference value, determined as the arithmetic mean of all the participants' results. If the deviation of a participant (marked as •) is within the range of uncertainty at 95 % level of confidence (which was self-declared by the participant and denoted by a bar), it is concluded that the national standard is equivalent to the other participants.

stability of traveling standards (laboratory standard microphones to be calibrated), analyzed calibration results of the participants, and developed the method to link the results of this international comparison to those of corresponding international comparisons by CCAUV<sup>[31]</sup>. Figure 8 shows results of the international comparison on pressure sensitivity of LS1P microphones. Figure 8 revealed that each participant's result agrees to the others within the uncertainty of the participant. Thus, mutual equivalence of acoustic standards among the participants was verified based on this data.

## 6.2 Validation of measurement capability of calibration service providers

NMIJ/AIST supported publishing a guide<sup>[32]</sup> for applying technical requirements given in ISO/IEC 17025 to the field of precise acoustic measurement. Calibration service providers established their quality systems by using this guide. Round robin tests were conducted to verify their measurement capability and NMIJ/AIST provided reference values as criteria. NMIJ/AIST also supported NITE from the technical viewpoint in the assessment of calibration service providers for accreditation. At the end of August, 2009, six calibration service providers were accredited as JCSS providers and their measurement capability was verified accordingly.

## 7 Research results

In this research, NMIJ/AIST made technical development indispensable to ensure the reliable environmental noise measurement and thus to sustain safety in our daily life. Firstly, primary calibration system of pressure sensitivities on laboratory standard microphones was advanced, resulting in a new calibration service of acoustic standards. Uncertainty caused by electrical characteristics of the calibration system was minimized to the limit at the present time. This revealed that instability of the microphone sensitivity is a dominant factor of uncertainty and that different types of microphones have different degrees of stability.

Secondly, NMIJ/AIST developed the method essential to evaluate the uncertainty related to the sound field. This evaluation has been an unsolved problem in secondary calibration of end-users' acoustic measuring instruments, conducted in comparison with laboratory standard microphones. Introduction of digital signal processing technique succeeded in decreasing the influence of indirect sound.

Lastly, the above technical development resulted in the new calibration service system of acoustic standards, ensuring traceability based on the Japanese Measurement Law. Measurement capability of calibration service providers was verified by several ways: supply of acoustic standards, discussion of the guide used to take technical requirements into account, provision of reference values in the round robin tests, and technical support in the assessment.

Besides calibration service providers, NMIJ/AIST was also assessed to get accreditation and took part in several international comparisons to verify the equivalence of national standards among the main institutes. NMIJ/AIST acted as a pilot laboratory in the first international comparison conducted in Asia and Pacific area.

These results greatly contribute to not only secure the performance of end-users' acoustic measuring instruments but also to improve the reliability of measurement by end-users. The method to evaluate indirect sound enabled the operator to find the cause and degree of indirect sound clearly. Thus, influence of indirect sound could be easily decreased by taking suitable measures and the effect of the measures could be quantitatively evaluated. As a result of the development, reliability of the measurement results became almost independent of the operator's skill and even an inexperienced operator could easily get reliable data.

## 8 Conclusion

NMIJ/AIST developed a calibration service system of acoustic standards based on the Japanese Measurement Law, with acoustic standards at the highest level of accuracy in the traceability system and started a new calibration service to meet the demands of the times.

The future theme is to expand the calibration frequency of acoustic standards outside of the audible frequency range. Equipment generating airborne ultrasound over 20 kHz is increasing around our living circumstances. However, sound pressure level cannot be quantitatively evaluated because acoustic standards are not yet established in the airborne ultrasound range. To discuss the problem of human safety under the exposure to strong airborne ultrasound, development of acoustic standards in high frequency range is essential<sup>[33]</sup>.

On the other hand, complaints against infrasound less than 20 Hz are increasing. Although common measurement procedures have been suggested for infrasound, reliability of the measurement results cannot be ensured without acoustic standards. Acoustic standards in low frequency range should also be essentially developed<sup>[34]</sup>. NMIJ/AIST is conducting research and development of acoustic standards and measurement technology necessary to expand the available frequency range.

## Acknowledgements

I am deeply grateful to Mr. Takeshi Fujimori, former staff of NMIJ/AIST and Dr. Sojun Sato, Professor of Akita Prefectural University and ex-staff of NMIJ/AIST as well as many persons concerned for much valuable advice.

## Terminology

Term 1. Sound pressure level: Sound pressure is normally expressed as sound pressure level because hearing ability of normal person ranges widely. Sound pressure level  $L_p$  is defined by the following equation:

$$L_p = 10 \log \frac{p^2}{p_0^2}$$

where  $p$  is rms value of sound pressure and  $p_0$  is reference sound pressure of 20  $\mu$ Pa which is minimal audible value for a sinusoidal signal of 1 kHz.

Term 2. Traceability: Traceability of a measuring instrument is ensured if the reasons for its uncertainty analysis can be deduced from the national standards.

Term 3. Pressure sensitivity: Pressure sensitivity is ratio of the open-circuit output voltage of the microphone to the sound pressure uniformly applied to the diaphragm.

Term 4. FFT analyzer: FFT analyzer is an instrument to calculate FFT (Fast Fourier Transform) of the input signal and it is useful for frequency analysis of acoustic signal.

Term 5. Wire meshed floor: Wire meshed floor is composed of crossed wires, tense enough for persons to walk on. Indirect sound can be decreased because sound wave passes through the squares of grillwork.

Term 6. Free-field sensitivity: Free-field sensitivity is ratio of the open-circuit output voltage of the microphone to the sound pressure that would exist at the position of the microphone in the absence of the microphone for plane progressive sound field.

Introduction of the microphone into the sound field changes sound pressure at the position of the microphone because sound wave is reflected or diffracted by the microphone. Free-field sensitivity of a laboratory standard microphone is essential for secondary calibration of acoustic measuring instruments to get precise sound pressure which is not influenced by the existence of the microphone.

Ratio of free-field sensitivity to pressure sensitivity depends on the shape of microphone housing and acoustic characteristics of the diaphragm. Thus specific type of microphones has particular value of the ratio. Measured ratio and its uncertainty are given for laboratory standard microphones<sup>[35]</sup>.

Term 7. Quality system: Quality system requires documenting the process of quality control based on the standard concerned to ensure the reliable calibration results. It consists of grounds for uncertainty analysis, practical calibration procedures, handling of instruments, personnel and calibration records.

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Completed the master's course in electrical engineering at the Faculty of Science and Engineering, Waseda University in 1989. Joined Electrotechnical Laboratory in 1990. Senior researcher, Acoustics and Ultrasonics Section, Acoustics and Vibration Metrology Division, NMIJ/AIST from 2001. Engages in research for precise acoustic measurement, especially for the development of acoustic standards. Studied at the National Physical Laboratory (NPL), UK for the development of infrasound standards from 2000 to 2001. Doctor of engineering.



## Discussion with Reviewers

### 1 Primary calibration method other than coupler reciprocity method

#### Question (Akira Ono, AIST)

In this paper, the coupler reciprocity method was used as a primary calibration method. Please introduce other methods if

any. What kind of methods is adopted for primary calibration by the other national metrology institutes which participated in the international comparison? If some institutes used methods other than the coupler reciprocity method, please explain the reason.

#### Answer (Ryuzo Horiuchi)

The free-field sensitivity of a laboratory standard microphone is used as a reference in the secondary calibration of acoustic measuring instruments and it is normally determined by multiplying a correction term to the pressure sensitivity which was obtained by using the coupler reciprocity method. Direct primary calibration of the free-field sensitivity by using the free-field reciprocity method is not practically used because the pressure sensitivity can be more precisely and easily determined than the free-field sensitivity.

In the free-field reciprocity method, two laboratory standard microphones are faced with each other in the anechoic chamber instead of the coupler's cavity and the voltage ratio is measured in the same way as the coupler reciprocity method. However, this method takes a long time for the measurement of voltage ratio and requires strict measures to decrease the cross-talk because signal-to-noise ratio deteriorates at the lower frequency range. Influence of indirect sound should be also minimized because the calibration is conducted in the anechoic chamber. These reasons prevent the method from being used as a routine calibration service. Thus the other national metrology institutes use the coupler reciprocity method as a primary calibration method.

There is the "laser-pistonphone" which is available as a primary calibration technique of the pressure sensitivity only for the lower frequency range. In this method, a piston attached to a shaker is used as a transmitter and generates sound pressure within the coupler. The vibration amplitude of the piston is optically measured and translated into the sound pressure. At the same time, output voltage of the laboratory standard microphone exposed to the sound pressure is measured and the pressure sensitivity is determined. NMIJ/AIST is developing a laser-pistonphone as a primary calibration system at infrasound range.

### 2 Methods used by other national metrology institutes for evaluating influence of indirect sound

#### Question (Akira Ono)

I think the method developed by NMIJ/AIST for evaluating influence of indirect sound in the secondary calibration is an excellent research result. Do other national metrology institutes adopt a similar technique or not?

#### Answer (Ryuzo Horiuchi)

Other national metrology institutes use methods different from NMIJ/AIST's to evaluate and minimize the influence of indirect sound in the secondary calibration of acoustic measuring instruments. TDS (Time Delay Spectrometry) eliminates indirect sound by using a narrow bandwidth filter which works considering the arriving time of indirect sound. MLS (Maximum Length Sequence) uses special random signals to obtain a pulse response rapidly. These methods have common characteristics of separating indirect sound from direct sound on the time domain. However, no methods have been internationally standardized yet. The FFT analyzer-based virtual pulse method developed by NMIJ/AIST will be adopted as one of the methods in the international standard under discussion.

### 3 International level of acoustic standards and Japanese way to go in the future

#### Question (Akira Ono)

Figure 8 seems to show little difference between the results of main institutes which participated in the international comparison organized by CCAUV and the results of institutes in Asia and Pacific area, from the point of view of uncertainty or deviation

from the reference value. It seems that, seen from another angle, Asia and Pacific institutes have smaller deviation from the average while some of the main institutes have larger deviation. Does this imply that calibration technique for acoustic standards became mature and it was transferred to the developing countries, resulting in technical equivalence among the countries? I would appreciate it if you could give me your view.

Furthermore, what will be necessary in the future for Japan to surpass the other countries from the point of view of reliability in acoustic measurement? Do you have any suggestions to the middle class of the traceability system (calibration service providers), to the lower class (end-users who conduct measurement at the site), or to the manufacturers of acoustic measuring instruments?

**Answer (Ryuzo Horiuchi)**

As the reviewer pointed out, the results of the international comparison (Fig. 8) show that there is little difference between the developing institutes in Asia and the leading institutes which have developed acoustic standards. This is due to the following reasons peculiar to acoustic standards. Many of the institutes in the world use the same type of primary calibration systems for laboratory standard microphones produced by one manufacturer of acoustic instruments. Calibration results can be reproduced once the operator becomes proficient in the calibration procedure. Technical information necessary for uncertainty analysis can be got without much difficulty. Therefore, even the institute with limited experience in microphone calibration can realize acoustic standards equivalent to those developed by the leading institutes. It is natural that calibration results obtained by using the same type of primary calibration systems have little deviation in the international comparison.

Just five institutes in the world (only NMIJ/AIST in Asia) have the advanced technology to develop primary calibration systems. The system developed by each leading institute has electrical or mechanical elements slightly different from other institutes. International comparison is the only way to validate their equivalence to the other institutes. It is concluded under the present technical situation that the international comparison shows good agreement among the institutes irrespective of calibration system specifications. Study of reasons for the remaining deviation would be necessary to improve the reliability of acoustic standards.

On the other hand, NMIJ/AIST is requested to take the technical leadership such as in the development of a new acoustic standard to surpass the other institutes from the point of view of reliable acoustic measurement. As described in the main text, our present theme is to expand the frequency range of acoustic standards. NMIJ/AIST is developing technical basis to sustain our daily life without any health damages caused by airborne ultrasound or infrasound.

At the same time, NMIJ/AIST should publish the research results by further study of measurement reliability from various viewpoints. In the international comparison, common calibration principles and traveling standards are adopted to validate the equivalence among the results of participants. Further consideration would be necessary to confirm the consistency among the results obtained by different calibration principles or by different types of acoustic standards. For example, influence of microphone types (LS1P or LS2aP) on the measurement results is not well evaluated yet.

Finally, as a suggestion to the calibration service providers, manufacturers and end-users of acoustic measuring instruments, I would like to appeal to them of certain necessary points to ensure the reliability of their measurement results. In addition to ensuring the traceability to national standards, uncertainty analysis on various components inherent in the measurement methods should be considered. I think the evaluation on the influence of indirect sound is a good example.

#### **4 International and societal role of NMIJ/AIST in the future**

**Question (Katsuhisa Kudo, Evaluation Division, AIST)**

Acoustic standards are essential in a lot of fields such as industrial, scientific and technological fields with wide-ranging end-users. Please tell us the international and societal role NMIJ/AIST should fulfill in the development of acoustic standards considering the technical trend.

**Answer (Ryuzo Horiuchi)**

As described in the main text, measurement of the noise from household appliances or information equipment has become important lately. Acoustic power level is mainly used to evaluate these noise sources instead of sound pressure level or sound level. Although traditional sound level intuitively gives noisy circumstances at the measurement points, acoustic power level can give total acoustic output generated by the noise source.

To ensure the reliable measurement of the acoustic power level, absolute calibration technique on a "reference sound source" should be established. It is a sound source specially designed for the precise measurement of the acoustic power level and it constantly generates noise with a wide bandwidth. The acoustic power level of the test sound source can be calibrated in comparison with the reference sound source whose acoustic power level is pre-determined. To secure the reliable measurement results for end-users, NMIJ/AIST is planning to develop precise calibration technique of reference sound sources and establish practical standards of the acoustic power level.

#### **5 Problem requiring most time to solve**

**Question (Katsuhisa Kudo)**

Please tell us the most difficult problem you have faced and the measures taken to solve the problem, ranging from the development of acoustic standards to the realization of the calibration service.

**Answer (Ryuzo Horiuchi)**

The most difficult technical problem in the development of acoustic standards was to discover the cause of instability in the pressure sensitivity of laboratory standard microphones. As described in the main text, the primary calibration system of laboratory standard microphones was advanced and uncertainty related to the electric circuit of the calibration system was minimized to the limit at present. After the improvement of the system, I got suspicious about the stability of the microphone sensitivity and studied the cause of the instability. In other words, the instability of the microphone sensitivity could not be observed until the system was advanced.

Possible causes of instability were physical distortion of the microphone described in the main text, sensitivity dependence on environmental conditions such as temperature and static pressure, poor insulation of the microphone, application of bias voltage necessary for the workings of the microphone, physical force applied to the microphone by its connection to the pre-amplifier. However, causes except the physical distortion of the microphone could not explain the measurement results.

The unstable phenomenon could not be observed by the simple repetition of the measurement. Usually the measured value has very small deviation but at a certain time it suddenly changes to an unexpected value. Quite a long time was spent to obtain a set of data under some measurement condition to confirm the stability. Besides the repetition of the measurement, measurement conditions were changed by trial and error. It took three or four years to come to the conclusion.

# Bioethanol production from woods with the aid of nanotechnology

## — Pretreatment for enzymatic saccharification using natural structure of cellulose —

Takashi Endo

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Bioethanol production from woody biomass by enzymatic hydrolysis of cellulosic components and fermentation has attracted much attention. In this process, pretreatment is important to improve enzymatic degradability of cellulose. A milling process is one of the most effective methods for pretreatment, but its high cost has been a problem. Recently we have developed the economically feasible wet-mechanochemical process as milling pretreatment, which can unravel cellulosic components into nanoscale fibers. Thus-obtained nanofibrous product showed a high enzymatic accessibility, while keeping the cellulose crystalline structure and the lignin content. This process is based on the understanding of the nanoscopic structural characteristics of wood and cellulose.

**Keywords :** Bioethanol, enzymatic saccharification, pretreatment, mechanochemical treatment, nanofiber

### 1 Introduction

Recently, the interest in bioethanol as automobile fuel has risen from the perspectives of measures for global warming and energy security. In the United States and Brazil, over 20 million kiloliters per year are produced. However, since the raw materials are food biomass such as corn and sugar cane, the increased price of foods and feedstuff in competition to the mass production of bioethanol has become an issue. Therefore, it is essential to establish a technology that allows use of nonfood biomass (cellulosic biomass) such as wood, straw, pasture grass, and others. Compared to the starch biomass such as corn, the cellulosic biomass is thought to be more effective in reducing carbon gas in terms of total life cycle assessment (LCA) from the production of raw material to the use of bioethanol<sup>[1]</sup>.

In general, bioethanol is produced by fermenting the sugar obtained from the raw materials, using yeasts. Therefore, it is necessary to hydrolyze (saccharify) the cellulose in wood to glucose. Currently, enzymatic saccharification method is drawing attention. Here, the pretreatment to enhance the enzyme reactivity of wood and cellulose is important.

Milling has been known as a highly effective pretreatment for enzymatic saccharification despite its high cost. Therefore, in developing the new pretreatment technology, by reinvestigating the milling process that is known to be effective, we clarified the mechanism by which milling enhanced the enzyme saccharification property of cellulose, using some new analysis methods. Next, we developed the new technology based on the findings obtained, and were able to construct an efficient and economically feasible pretreatment technology based on the new concept.

### 2 Knowing wood and cellulose

The pretreatment technology for enzymatic saccharification that we developed is a method that utilizes the structural characteristics of wood and cellulose. Therefore, the structures of wood and cellulose will be outlined since they are important for developing this technology.

#### 2.1 The basis of cellulose

The main components of wood are cellulose, hemicellulose, and lignin. The molecules of cellulose and hemicellulose are composed of sugars while lignin is a complex aromatic compound. In wood, the percentage of cellulose is highest at about 40~50 %. Cellulose is a biological macromolecule where the glucose is chain-linked, and the nature of cellulose in wood is an assembly of cellulose molecules called the cellulose microfibril. When the cellulose molecules are biosynthesized, they self-assemble regularly like stacked boards to form cellulose microfibrils of 3~5 nm width (Fig. 1, right bottom). This microfibril is the main body of the cellulose crystal, and it does not dissolve in water or ordinary organic solvents since it is extremely stable. However, the assembly forces of the cellulose molecules are the intermolecular force and the hydrogen bond that are generally considered weak. Although the component sugar is glucose, amylose in starch has different chemical and physical properties from cellulose and dissolves in hot water. Therefore, enzymatic saccharification by amylase occurs quickly, and bioethanol can easily be produced.

#### 2.2 Wood structure is nanostructure

As shown in Fig. 1 right top, the “wood fiber” is composed of nanosize “cellulose microfibrils” held together using hemicellulose and lignin as adhesives, and the “wood structure” is formed by laying the vessels and tracheids that

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carry water in the middle and then layering the microfibrils around them (Fig. 1 left top). The toughness of wood results from this layer structure. This is often likened to a barrel or tub. A tub is strong because the boards are arranged circularly in a vertical direction, and the hoop is used to bundle them together in a direction 90 degrees of the boards. In the wood structure, the nanosize cellulose microfibrils act like the boards and hoops of a tub, and form a highly layered nanostructure. This tough wood structure makes the pretreatment for enzymatic saccharification very difficult.

### 3 Issues in conventional technology and scenario for the development of new technology

#### 3.1 Acid saccharification and enzymatic saccharification

The method for saccharification of cellulose in the wood can be divided roughly into acid and enzyme methods. Figure 2 shows the advantages and issues of the two methods. The oldest method used is the acid saccharification using sulfuric acid. Currently, large-scale bioethanol manufacturing experiments incorporating new technologies is in progress for this method. The greatest advantage of the sulfuric acid saccharification is that the reaction occurs in a short time using inexpensive sulfuric acid as the catalyst.

However, the facilities must be resistant to sulfuric acid, and the recovery and removal of sulfuric acid from the saccharified solution and waste liquid are remaining issues. While these can be solved with the advancement of engineering technology, the greatest problem is that the produced sugar is susceptible, in principle, to over-decomposition into furfural by the action of coexisting sulfuric acid<sup>[2]</sup>. When over-decomposition occurs, the yield

of sugar that enables ethanol fermentation is reduced, and the product of over-decomposition even at relatively small amounts (few %) may inhibit yeast fermentation.

On the other hand, since, in principle, no side reaction occurs in enzymatic saccharification, the yield of ethanol end product is expected to increase. The enzymatic reaction progresses at mild conditions of about 50 °C, and the environmental load is low since it does not require large amounts of chemicals. The enzymes for the saccharification of cellulose are generally called cellulase. There have been over 500 kinds identified, and reactivity of some of them is affected greatly by the crystallinity and structure of cellulose. In actual enzymatic saccharification, a mixture of several enzymes including the ones for hemicellulose is used rather than a single species of enzymes.

However, there are also issues in enzymatic saccharification. Enzymatic saccharification requires much longer time compared to acid saccharification. Also, while cellulase is used in the food and textile industries, it is actually fairly expensive. As mentioned earlier, in some cases, large quantity of enzyme is required because the enzymatic reaction may be affected by the structure of cellulose.

#### 3.2 Pretreatment technology for enzymatic saccharification

Since untreated wood does not react with enzymes, it is necessary to conduct the pretreatment to increase the reactivity of wood and cellulose and to promote enzymatic saccharification. According to the empirical rules of pretreatment gathered from past R&Ds, the points considered were: to increase of surface area by breaking up the wood into small pieces; to increase reactivity by amorphization of highly crystalline cellulose; and to breakdown and

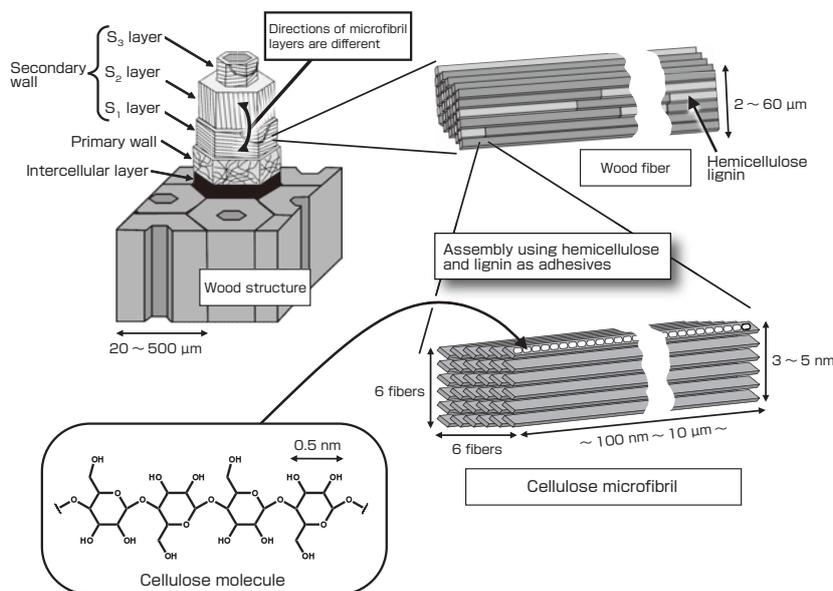


Fig. 1 Diagram of cellulose microfibril and wood structure.

remove the lignin component that may inhibit the enzymatic activity. The pretreatments according to these rules include milling, cooking, and steam explosion<sup>[3]</sup>, but all methods have issues (table in lower part of Fig. 2). In the milling treatment, equipments such as the ball mill are used. The operation is simple and the enzymatic saccharification degree of the pretreated material is high. However, the power consumption is high and processing efficiency is low since it is batch processed. In many cases and the cost turns out high. Cooking treatment is a method where the lignin and hemicellulose in the wood are decomposed and removed using chemicals and water. It is similar to the pulping process of papermaking. Although the saccharification degree of the treated material is high, the dependency on certain wood species and the waste liquid are issues. In the steam explosion treatment, the material is placed in high-temperature high-pressure steam for a certain time, exposed to atmospheric pressure immediately, and the wood is broken down into fibers by the rapid volume expansion of steam. The need for high-pressure resistant facilities, heat recovery, and dependency on wood species are issues. Recently, hydrothermal treatment using pressurized hot water over 100 °C has drawn attention. This method uses the hydrolysis action of hot-compressed water, but like in the acid saccharification, over-decomposition tends to occur at high temperature of 200 °C or over.

Therefore, we conducted re-experiments for the milling treatment, which is a simple yet highly effective operation, and analyzed and evaluated the mechanism for increased saccharification from the perspectives of wood chemistry and cellulose chemistry. Moreover, based on the findings,

we attempted the development of the optimal pretreatment technology by combining several technologies.

#### 4 Mechanochemical treatment technology

We conducted R&D on technologies to pulverize the cellulose materials such as pulp, and technologies to composite pulp or wood powder with resin, by controlling the aggregation of particles produced by milling (control of formation of hydrogen bonds)<sup>[4]-[7]</sup>. In the process of pulverization of materials in the milling process, reactions such as breaking of chemical bonds occur. Also, the pressure and shearing force of milling can form bonds and synthesize new materials. Since milling causes the chemical reaction by the mechanical process, it is called the mechanochemical treatment. We consider the formation and breaking of bonds including weak bonds such as the hydrogen bond and the hydrophobic bond (intermolecular force) as part of the mechanochemical treatment. The mechanochemical treatment was developed originally as the technology for compositing of inorganic substances and metals, and as alloy technology. It is possible to form covalent bonds such as esterification reaction in organic substances. However, there is a “threshold value” for the change of substance to occur in milling, and breakdown and compositing will not occur even if milling is done for a long time unless enough milling energy that can set off the chemical reaction is applied.

As explained so far, the mechanochemical treatment is a relatively simple mechanical process, and has been known to improve the enzymatic saccharification of cellulose<sup>[8]</sup>. The important points were considered to be milling the wood

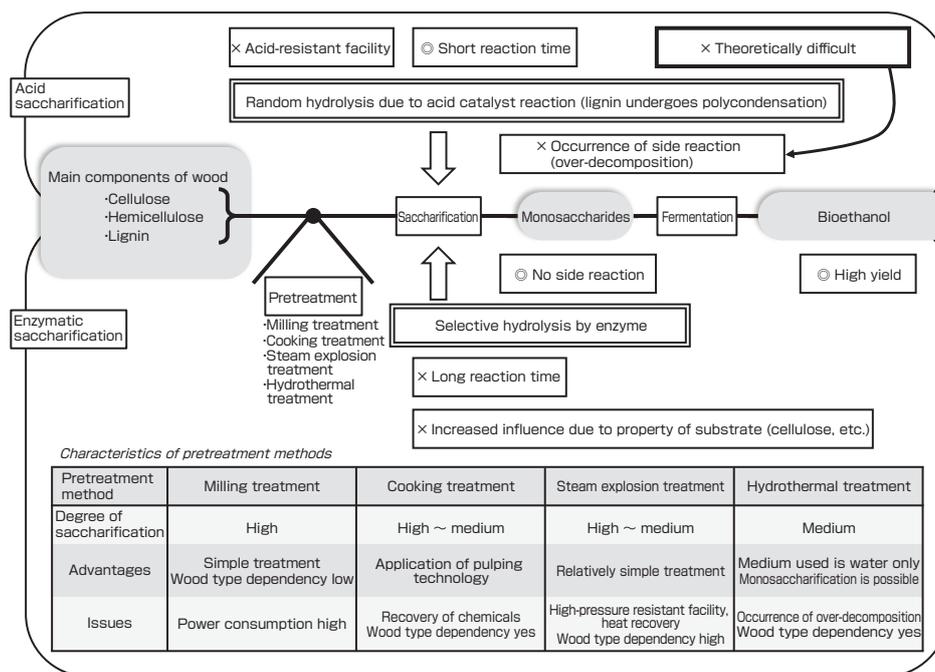


Fig. 2 Characteristics of acid saccharification and enzymatic saccharification methods.

materials thoroughly in the dry state using equipments such as the ball mill to produce fine wood powder, and to destroy the crystal structure of cellulose (amorphization).

Consideration of the mechanism for improving the saccharification by such mechanochemical treatment from the perspective of size, the process is as follows. The saccharification reaction of cellulose by cellulase is the breaking of the bond between glucose with length of 1 nm or less at the active site of cellulase, a protein. As a first step, cellulase must adhere to cellulose. This means cellulase first adheres to the microfibril, since cellulose is basically a microfibril. Cellulase is about 5 nm as a sphere, but the wood powder obtained by dry milling is much bigger. The wood powder obtained by the general dry mechanochemical treatment is about 10 μm even if the processing condition is optimized, and hardly any submicron or nanosize wood powder is produced even by prolonged processing<sup>[9]</sup>. The reason is because the aggregation of particles occurs when the wood is finely broken down in the mechanochemical treatment, and the particle size may increase depending on the material.

Another point from the size perspective is the crystallinity of cellulose. The evaluation is done in most cases by powder x-ray diffraction. As mentioned in section 2.1, the cellulose crystal is a microfibril, and the amorphization of cellulose is simply looking at the disarrangement of the hydrogen bond with length of 1 nm or less in the region of 3~5 nm or less. The change of cellulose from crystal to amorphous is a change in a smaller region, considering the size of cellulase. While

there are many reports on the enzymatic saccharification of cellulose, the reaction mechanism from the size perspective of cellulose and cellulase had not been clarified.

Figure 3 is a summary of the points of the conventional pretreatment technologies from the size perspective. According to the knowledge so far, the destruction and fiber production of wood structure at the level of tens of μm, the compositional change by decomposition and removal of hemicellulose and lignin, and the change of crystallinity at the nano level were considered important. There were hardly any discussion for the several nm region at the initial phase of the enzymatic saccharification reaction (adsorption of cellulase to cellulose microfibril), and there was not much viewpoint from the microfibril side.

From the above, concerning the factors that improve the enzymatic saccharification of wood through mechanochemical treatment, we thought some guide can be obtained to construct the optimal pretreatment technology by carefully analyzing from various perspectives including the analysis at the microfibril level.

#### 4.1 Re-investigation of classic technology

For the mechanochemical treatment of wood, we conducted basic experiments using a planetary ball mill, and investigated the relationship between the physical and chemical properties and the saccharification of the product<sup>[10]</sup>. When the mechanochemical treatment was conducted using roughly milled hardwood powder (Eucalyptus < 0.2 mm), the breakdown of raw material wood powder progressed but

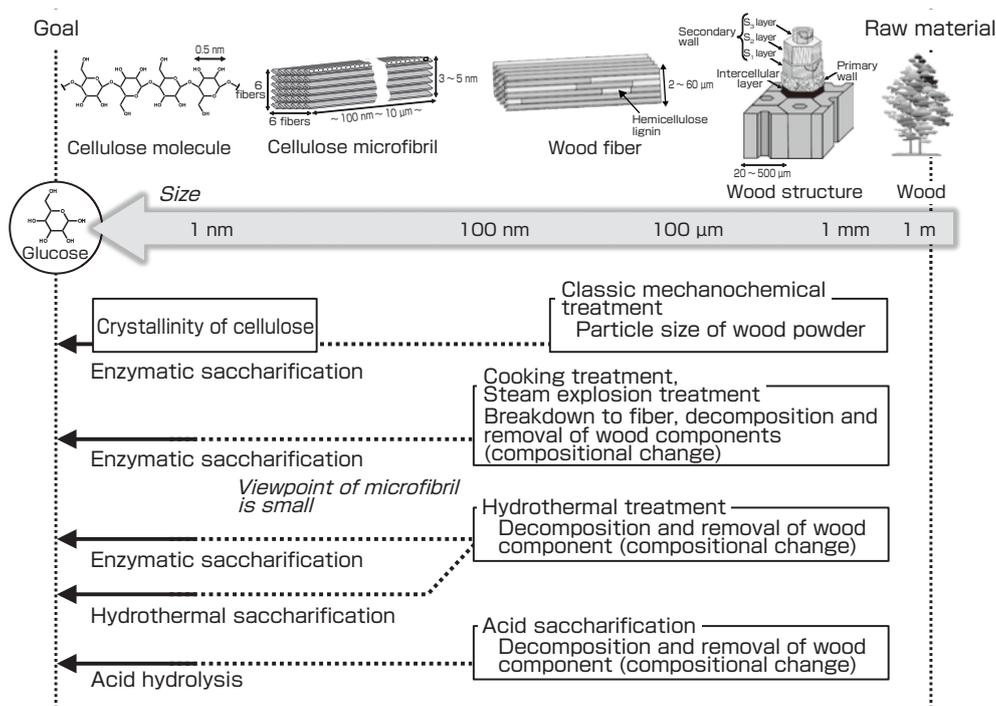


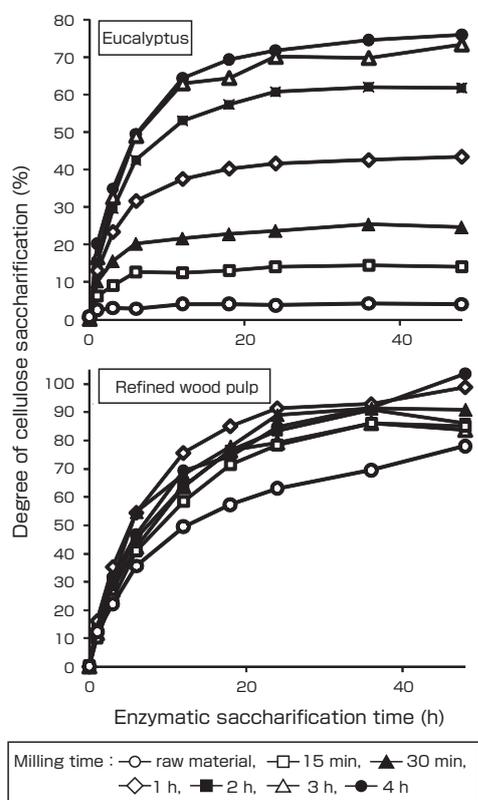
Fig. 3 Size image of (conventional) pretreatment technology for enzymatic saccharification.

the average particle diameter of the product ceased to change from about 20  $\mu\text{m}$  even after milling for 1 hour or more. This indicated that the equilibrium value for the particulation and aggregation of the particles was about 20  $\mu\text{m}$ . Looking at the enzymatic saccharification of the product, the saccharification degree was extremely low even though the untreated raw material was of fine powder of 0.2 mm or less. However, saccharification increased as the time of mechanochemical treatment increased. After four hours, the saccharification was 20 times or more compared to the raw material. Similar trend was seen for Douglas fir, a softwood. However, major difference in results was observed when the mechanochemical treatment was conducted using refined wood pulp (fibrous, width about 20  $\mu\text{m}$ , length about 200  $\mu\text{m}$ ) in a comparative experiment. In pulp, saccharification was high even in the untreated material, and the effect of mechanochemical treatment was small (Fig. 4). From these results, it was found that enzymatic saccharification property could not be estimated from the size of the material alone.

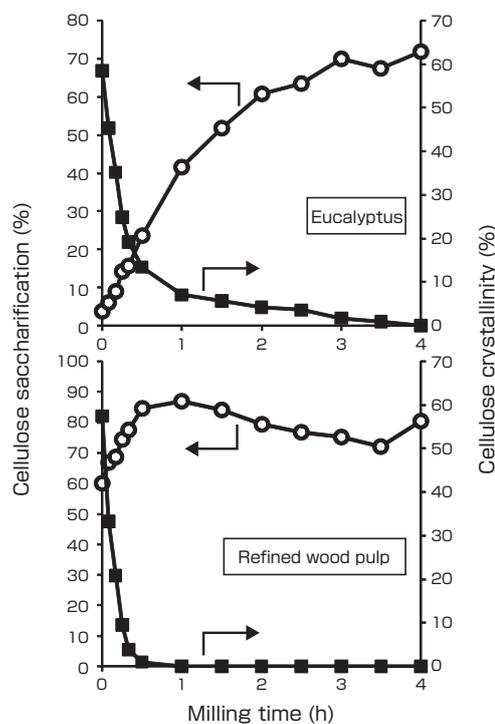
Next, the relationship between the crystallinity of cellulose and saccharification was investigated by x-ray diffraction. As a result, in case of wood, crystallinity decreased rapidly with mechanochemical treatment whereas saccharification increased gradually. On the other hand, in the case of pulp, saccharification was high even though the raw material had high crystallinity, and crystallinity decreased rapidly by mechanochemical treatment just as in wood, but the

relationship between crystallinity and saccharification was low (Fig. 5). The results of the mechanochemical treatment experiment conducted using woods such as Eucalyptus as raw materials did not greatly contradict the previous understanding that amorphization of cellulose was important for enzymatic saccharification. However, when pulp was used as the raw material, high saccharification was obtained with highly crystallized material, and there was a contradiction. The phenomenon where the enzymatic saccharification gradually decreased despite being amorphous when amorphized cellulose was processed for a long time by mechanochemical process had been known for some time.

From the above, it was shown that the enzymatic saccharification could not be sufficiently explained by the particle size of the mechanochemically treated product or the crystallinity of cellulose. In the above experiments, enzymatic saccharification was conducted without purification of the mechanochemically treated products, but the enzymatic saccharification degree was high. This means the enzymatic saccharification progressed even when the wood components such as lignin that supposedly inhibit enzymatic reaction remained. Moreover, from other experiments, the lignin component had high molecular weight as in an untreated wood structure even after undergoing mechanochemical treatment. Also, solid-state nuclear magnetic resonance (NMR) measurement and infrared spectrometry showed that there was hardly any transformation of wood components such as oxidation by the mechanochemical treatment.



**Fig. 4** Change in degree of enzymatic saccharification by milling time.



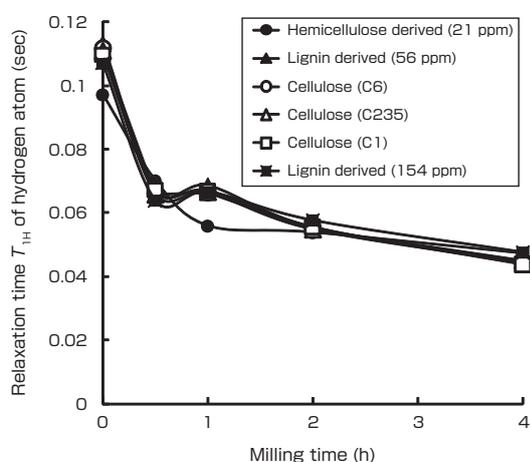
**Fig. 5** Relationship between degrees of crystallinity and enzymatic saccharization.

#### 4.2 Application of macromolecular chemistry techniques

Since the aforementioned experiment results indicated that the fine breakdown of wood, amorphization of cellulose, and separation of lignin, which were the empirical rules of conventional technology, were not important in increasing the enzymatic saccharification of wood, analysis from a new viewpoint became necessary.

NMR is used frequently as an analysis tool of molecular structure in the fields of organic chemistry, and in the macromolecular field, the evaluations of mixture of molecules in complexes and molecular aggregation size (domain size) are conducted by relaxation time measurement using solid-state NMR. Relaxation time measurement is a method in which pulse signals are irradiated to a substance using the NMR device, and how fast the signal decays is measured. If the aggregation (domain) of the substance is large, the pulse signal takes more time to travel far. If the domain size is small, the pulse signal decays fast. In a case where different substances are mixed at the molecular level and placed in the same environment, the different substances will have the same relaxation time. The result of using this method to evaluate the mechanochemically treated product is as follows.

Solid-state NMR measurement was conducted at a similar moisture condition as the enzymatic saccharification reaction, and the relaxation time ( $T_{1H}$ ) of hydrogen atoms of the cellulose molecules was measured. As a result, the relaxation time decreased (the molecular motion increased in the macromolecular substance such as cellulose) with more mechanochemical treatment, and it was 0.05 sec. after 4-hour milling when saccharification reached maximum (Fig. 6). When the domain size was calculated from this value, it was 5.5 nm<sup>[11]</sup>. The wood became wood powder of about 20  $\mu\text{m}$



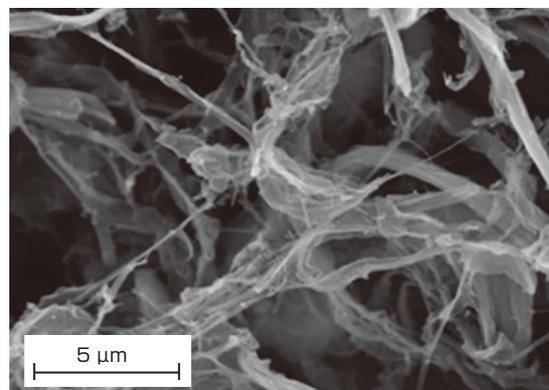
**Fig. 6** Change in relaxation time  $T_{1H}$  by milling time (figures in parenthesis are assignments of wood components in NMR).

in appearance by the mechanochemical treatment, but this wood powder is actually composed of domains of about 5 nm, which is much finer. Since this 5 nm size approached the width of the cellulose microfibril, it was hypothesized that the major factor for the promotion of enzymatic saccharification was that the microfibrils separated from each other through the mechanochemical treatment, thereby increasing the surface area where the enzymes can adsorb. The studies hereafter were based on this working hypothesis to clarify the effective mechanochemical treatment for enzymatic saccharification, and we developed a new pretreatment methodology. While the cellulose became amorphous by the mechanochemical treatment, it is thought that the orientation of cellulose molecules such as microfibril remained<sup>[12]</sup>. Looking at the size level of the fibril separation, the cellulose crystallization occurs at a lower level, and whether it is high crystallinity or amorphous is not particularly important.

#### 5 Microfibril treatment

The enzymatic saccharification reaction of cellulose is a solid-liquid reaction of solid cellulose and enzyme dissolved in water. In general, to promote the solid-liquid reaction efficiently, the solid can be broken down finely to increase the contact surface with the liquid. The microfibril in the wood is the minimum unit of solid cellulose. Saccharification is expected to progress smoothly if the cellulose can be dissolved and separated into one unit of molecule, but the enzyme will easily become deactivated in a solvent that can dissolve cellulose.

Therefore, to confirm the working hypothesis based on the results obtained from the aforementioned solid-state NMR measurement, we investigated the method for actually unraveling the microfibril, which is a microscopic wood fiber. In the papermaking technology, the process called “beating” is done to increase the strength of the paper. In this process the fibers are fluffed by mechanically applying



**Fig. 7** Scanning Electron microscope photograph of wet mechanochemically-treated product.

shear force to the water-dispersible slurry of pulp. The water enters the minute gaps of pulp fiber by shear force, acts like wedges to unravel the fibers, and the surrounding water prevents the aggregation of the fluffed fibers. We thought the wood could be broken down to microfibrils by using some treatment similar to the beating process. Therefore, as basic experiment, wet mechanochemical treatment was done using the ball mill with wood powder dispersed in water at 20 times the quantity by weight, and we obtained some highly viscous, creamy product. The product was dried and observed under the scanning electron microscope. It was found that the microfibril formation progressed, and fine fibers of 100 nm or smaller, some of 20 nm at the narrow parts, were produced (Fig. 7). The degree of cellulose crystallinity was studied by x-ray diffraction, and it was found that the same degree of crystallinity was maintained as the raw material. Since the microfibril itself is the crystal body of cellulose, the retention of crystal structure indicated that the microfibril could be unraveled without much damage. Although this wet mechanochemical product (microfibrilized substance) had high crystallinity, degree of saccharification was 70 % or above. This result showed that decreasing the crystallinity of cellulose was not important to increase the enzymatic saccharification, but it was more important to increase the surface area with which the enzymes could react by separating the microfibrils from each other. While the concentration of solids was about 5 % in the wet mechanochemical product, the cellulose microfibril with specific gravity of 1.5 was dispersed without settling out as the water molecules were retained around it. Therefore, space where the enzymes could act freely was formed around the microfibrils. Since the main residue of the saccharification experiment were large fiber structures in the samples with insufficient wet treatment and low saccharification, it was found that the enzymatic saccharification could be increased greatly if the wood were thoroughly broken down into microfibrils.

As presented above, the working hypothesis that the separation of microfibril was effective for enzymatic saccharification based on the solid-state NMR, was proven by actually creating microfibrils from wood by the wet mechanochemical treatment. The crystallinity of cellulose was not important in this pretreatment. In section 4.1, the refined wood pulp had high degree of enzymatic saccharification while it was highly crystallized, but in this case, it was thought that the microfibrils separated from each other in the refining process of breaking down and removing the hemicellulose and lignin, and the surface to which the enzymes could approach and react increased. The wood structure is an assembly of microfibrils, and the assembly force is a weak bond such as a hydrogen bond. Therefore, by using the water molecule like a wedge in the wet mechanochemical treatment to break the hydrogen bonds between the microfibrils that keep them together,

the microfibril building blocks can be disassembled easily. This method is not troublesome from the wood chemistry perspective.

## 6 Microfibrilization technology for practical application

### 6.1 Investigation of continuous and mass treatment method

The ball mill used in the fundamental experiment for the microfibrilization treatment is useful for experiments with small amounts of samples, but is done as a batch process, so it is not practical in terms of upsizing and cost reduction. Therefore, we considered a treatment method where shear force and pressure could be applied to the raw material as a wet method. We decided continuous and mass production would be possible using a disk mill that had a similar milling mechanism as a stone mortar.

We used a disk mill (Supermasscolloider, Masuko Sangyo Co., Ltd.) to repeatedly mill the wood powder slurry (wood powder concentration 5 wt%), and found that the microfibrilization progressed as in the ball mill, and the enzymatic saccharification of the product greatly increased. In the disk mill treatment, the upper and lower disks were set at about 10  $\mu\text{m}$  distance, and the processing efficiency was 10~20 times or higher than the ball mill. However, in the disk mill, there was a dependency on the wood species that was not significant in the ball mill. In some cases, the hardwood did not microfibrilize sufficiently and the saccharification did not improve compared to softwood. This was because the milling energy was smaller in the disk mill compared to the ball mill. Therefore, treatment to weaken the wood structure was necessary before the disk mill treatment.

### 6.2 Combination treatment for weakening the wood structure

The origin of wood strength is the robust layer structure as mentioned before. Although the microfibrils are held together by weak bonds such as hydrogen bonds, separation to microfibril units is not easy due to its board-and-hoop layer structure. Therefore, we thought the wood structure would weaken and the disk mill treatment could be conducted effectively by first destroying the structure that corresponded to the hoop of the tub to increase the water permeability, and then removing the hemicellulose that glued the microfibrils together.

The method for destroying the structure that corresponded to the hoop was preliminarily investigated using the ball mill. After conducting dry mechanochemical treatment of the raw wood powder for a certain time, water was added for wet treatment, and the microfibrilization and saccharification of the product were studied. As a result, when the time of dry mechanochemical treatment was 15

min or less, no major change was seen, while saccharification significantly improved in the wet-treated product obtained after dry treating for 20 min or more. When the dry mechanochemically treated product was observed by a scanning electron microscope (SEM), it was found that the large wood structure of the raw wood powder was almost entirely destroyed by the 20-min dry mechanochemical treatment. Since the ball mill process was not practical for destroying the hoop structure, we considered the method that had relatively large milling energy and enabled mass processing, and found that wet cutter mill was effective. The wet cutter mill we employed (Micro-Meister; Masuko Sangyo Co., Ltd.) had powerful shear force with a fixed blade and a rotary blade that rotated at ultra high-speed of 10,000

rpm or more, and was capable of instantly breaking down the raw material dispersed in water to 1 mm or less. In this case, water fluidized the milled product, prevented stagnation, and promoted the break down efficiently. Also, the water that permeated the wood structure during the process also worked effectively for the following autoclave and disk mill treatments.

Next, we conducted the hydrothermal treatment using the autoclave as a method for reducing the adhesion effect of hemicellulose. The hydrothermal treatment allowed the selective hydrolysis of hemicellulose component according to the temperature condition<sup>[13]</sup>. Investigating a combination treatment method that combined the wet cutter mill and autoclave treatments, it was found that efficient pretreatment was possible regardless of the wood species by conducting the autoclave treatment (135 °C) after conducting the wet cutter mill treatment (1 mm or less) on the roughly milled raw wood powders, and then conducting the disk mill treatment as the final stage. When the Eucalyptus raw material was roughly milled to 3 mm or less by the combined treatment, the degree of saccharification was 4 times or more compared to the case when wood powder 0.25 mm, which was finer, was treated with the disk mill only. Approximately the same saccharification was obtained as for the long dry ball milling (Fig. 8).

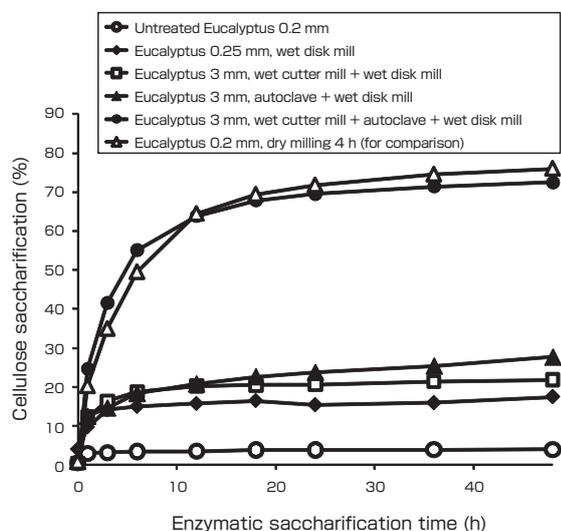


Fig. 8 Increasing efficiency of disk mill treatment by combination treatment.

### 6.3 Mechanism of improvement of enzymatic saccharification by combination wet mechanochemical treatment

The mechanism by which the enzymatic saccharification improves due to the aforementioned combination wet mechanochemical treatment is thought to be as follows

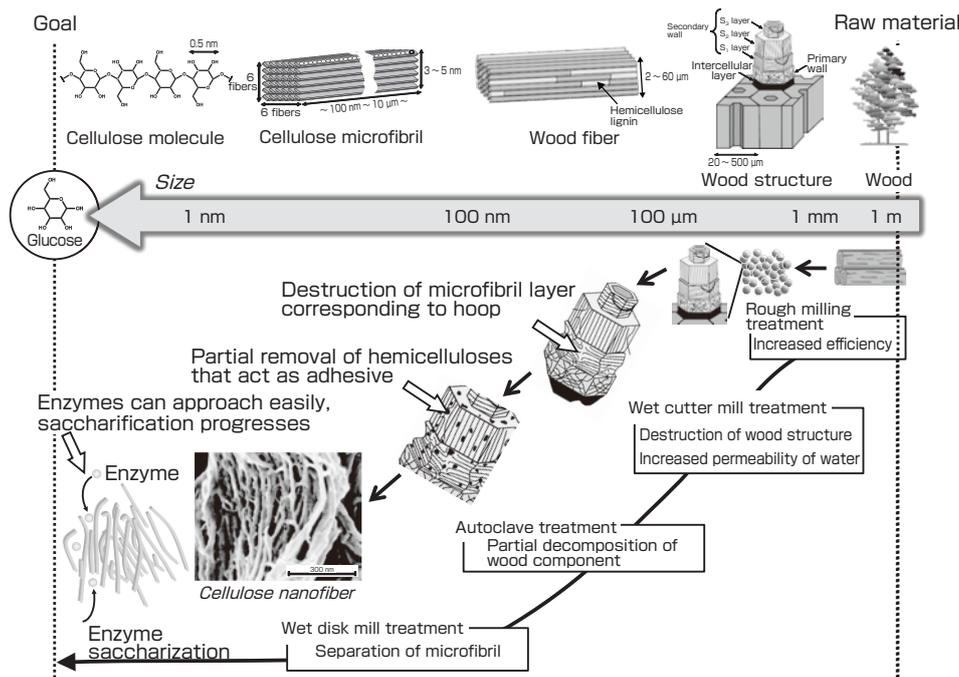


Fig. 9 Size image of combination wet mechanochemical treatment.

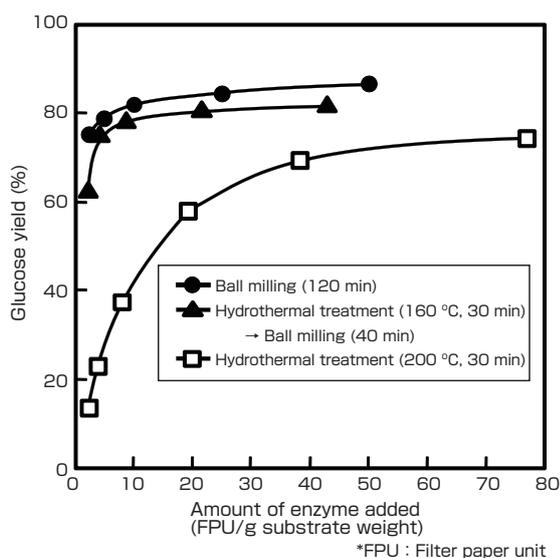
(Fig. 9). The cell structure of wood is 20-500  $\mu\text{m}$ , and the wood structure is destroyed by the powerful shear force of the wet cutter mill. The microfibrils that correspond to the hoop are partially broken. Next, the hemicellulose that glues the microfibrils together is partially hydrolyzed (few % of all hemicelluloses) by the autoclave treatment. When the autoclaved product was observed at high magnification by SEM, a large number of pores of tens of nm were observed. These pores are thought to be the holes after the hemicellulose dropped out. As the wood structure weakens after these steps, the structure can be unraveled into microfibrils easily by the final disk milling, and the enzymatic saccharification is improved greatly.

So far, the explanation was centered on the mechanism for the improvement of enzymatic saccharification of the cellulose component. However, in bioethanol manufacturing, the saccharification of hemicellulose is also important to increase the yield of sugar as fermentation material. In the wood structure, hemicellulose covers the surface of the cellulose microfibril, and in our pretreatment process where the microfibrils are separated from each other, the saccharification of hemicellulose progresses alongside the saccharification of cellulose. Since drastic chemical reaction does not take place in our pretreatment, the structure of lignin does not change from the structure in the untreated wood. After enzymatic saccharification, the lignin remains as residue.

## 7 Advantage of the mechanochemical treatment

### 7.1 Cost reduction of enzymatic saccharification

As mentioned earlier, the disk mill treatment has high efficiency and relatively low cost. However, compared to the



**Fig. 10 Relationship between amount of enzyme added and saccharification by different pretreatment.**

simple heat treatment, dramatic cost reduction is not possible since the driving power of motors cannot be recovered or recycled. Yet, the advantage of the mechanochemical treatment is that the costs in other processes of the bioethanol production can be reduced greatly. In the mechanochemical treatment, enzymatic saccharification and fermentation progress efficiently without the chemical treatments such as lignin removal. Also, dependency on raw material is low, and the process can be applied as the pretreatment of hardwoods, softwoods, straws, and others. The greatest advantage is the reduction in the cost of enzymes such as cellulase, which in some cases dominate over half the cost of bioethanol production. The mechanochemically treated products can be sufficiently saccharified with relatively small amount of enzymes. Figure 10 shows the relationship of the amount of enzymes and the degree of saccharification of the products of various pretreatments<sup>[14]</sup>. Compared to the mechanochemical treatment, the degree of saccharification falls greatly when the amount of enzymes is small in the 200 °C high-temperature hydrothermal process. This phenomenon is thought to be due to the production of inhibitor from the transformation of wood components in the high-temperature hydrothermal process. Similar results are expected in the acid treatment that follows the similar reaction as the hydrothermal treatment. Relatively low temperature (160 °C) treatment reduces the time for mechanochemical treatment, and saccharification progresses even with small amount of enzymes.

### 7.2 Comparison with conventional technology

The comparison of the characteristics of the conventional technology against the pretreatment process for enzymatic saccharification we developed is as follows (table at bottom of Fig. 2). The degree of saccharification of the treated product is about the equivalent to that of the classic mechanochemical treatment. Advantages are: there is no need of advanced reaction control as in the chemical process; and since only water is used for the process, recovery of chemicals is not necessary and waste liquid management is easy. With the same amount treated, the electricity consumed by our process is about 1:10~20 or less of the conventional ball mill process. The autoclave treatment also has low energy consumption since it is not a high-temperature high-pressure process, and the equipment does not have to be high-pressure resistant. As mentioned earlier, the dependency on raw material biomass species is low. Also, our pretreatment process is basically a wet process, and the raw material does not have to be dried. Raw materials with high water content can be used as is. In the concentrated sulfuric acid saccharification, it may be necessary to dry the raw material to prevent heating due to dilution of the sulfuric acid, and therefore, the efficiency is poor.

The pretreatment process we developed overcomes the various issues of the conventional technology, and can be applied without losing the advantages. The new issue is

that since the wet cutter mill and the disk mill used in this R&D are precision mills, they cannot be readily upsized. However, since our pretreatment process is similar to the mechanical pulping technology of papermaking, we believe up-scaling and practical utilization can be done by applying and developing the papermaking technology.

### 8 Summary and future developments

At AIST, we built an integrated manufacturing mini-plant (capable of treating 200 kg of diverse biomass at once) that incorporates our pretreatment process and the saccharification and fermentation processes. Here, extraction of issues in individual elemental technologies and process continuity, issues on biomass species, and economical feasibility assessments are being done. Currently, R&Ds are conducted with companies for commercialization using large-scale processes, and papermaking technologies are being incorporated.

In the practical application of bioethanol production technology, high added value for residues and byproducts is also important. The microfibrilized product obtained by the pretreatment of wood is called cellulose nanofiber since it is a nanosize fine fiber. Compared to steel of the same size, its weight is 1/5 lighter and the strength is 5 times greater. Currently, the development of lightweight high-strength material using this property is being developed<sup>[15]</sup>. Also, application to optical materials and cosmetics is being studied. Some are already in use as filter materials and food additives.

In our pretreatment process, no major change in the molecular structure of the wood components occurs. Therefore, the lignin residue after the enzymatic saccharification is unlike the black liquor (broken down lignin) produced as waste in the papermaking process. It is thought to contain structures similar to wood with high molecular weight. This lignin residue can be converted to polymer material and high value-added material that was impossible with lignin in the black liquor that could only be used as fuel. Extremely high economic feasibility can be attained using the cellulose nanofiber production as a common fundamental process with the production of bioethanol (main) and high value-added material (subsidiary).

Figure 11 is a summary of the flow of our pretreatment technology development. As a result of studying the conventional technology, we found that the viewpoints from the wood chemistry and cellulose chemistry of the target wood were necessary for the development of the practical pretreatment technology. Moreover, by combining the knowledge and technologies of other fields such as macromolecular chemistry and papermaking, we were able to construct a pretreatment technology based on a new concept. However, there are many unclear points about the properties and reaction mechanism of the enzymes for the cellulose nanofiber obtained by pretreatment, and state-of-the-art analyses are necessary. In the future, the technologies and knowledge of biotechnology, chemical engineering, thermal engineering, LCA assessment, and social sciences may be fused to establish a practical biofuel technology.

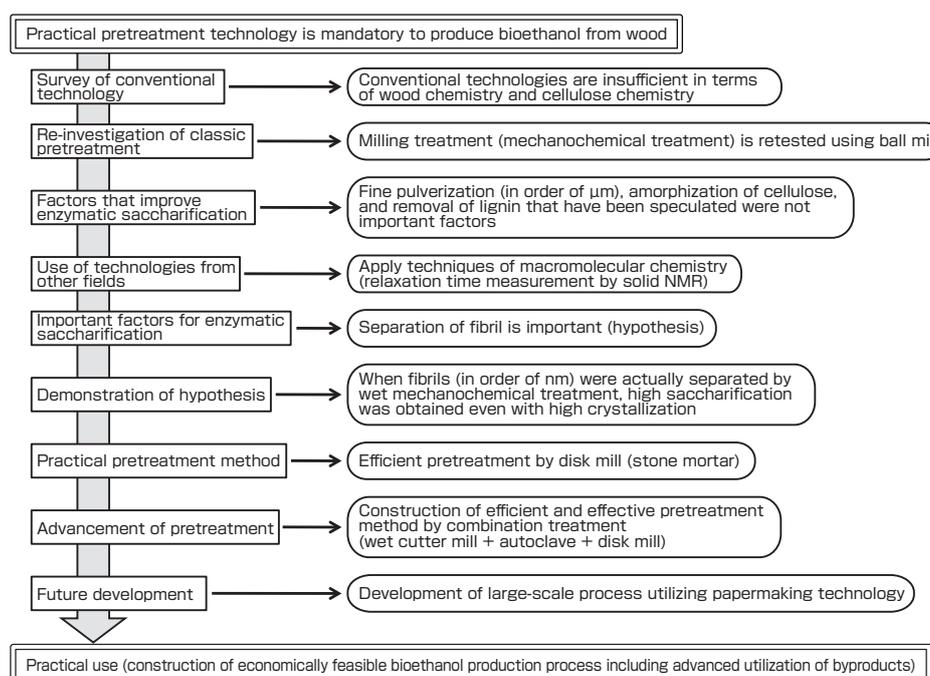


Fig. 11 Flow of pretreatment technology development.

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## Author

### Takashi Endo

Completed the doctorate course at the Department of Chemistry, Graduate School of Science, Hiroshima University in 1992. Doctor (Science). Joined the Shikoku National Industrial Research Institute, Agency of Industrial Science and Technology in April 1992. Engaged in research for the pulverization of cellulose and wood using the milling technology and high added-value technology. Worked mainly in the field of materials to 2003. After transferring to the current institute in 2004, has conducted R&D for bioethanol. Leader of the research team of the Biomass Technology Research Center from 2006 to present. This paper is a summary of part of the research results for cellulose milling that has been the subject of study since joining the institute.



## Discussion with Reviewers

### 1 Advantages compared to conventional treatment methods (cooking, steam explosion, and hydrothermal methods)

#### Question (Koichi Mizuno, Research Institute for Environmental Management Technology, AIST)

In the conversion process from wood to ethanol when limited to enzymatic saccharification, Fig. 2 shows the comparison of the developed method combining the wet cutter mill, autoclave, and wet disk mill treatments to other conventional methods (cooking, steam explosion, and hydrothermal treatments). Please indicate quantitatively or qualitatively how much the advantages and strengths have been progressed and to what degree the disadvantages and problematic issues have been overcome.

#### Answer (Takashi Endo)

The advantage of the combination treatment we developed is that a pretreated product with high degree of saccharification can be obtained with low environmental load and with no dependency on wood species. Compared to ball milling, the conventional technology, the consumption energy is 1:10~20 or less. Also, in our pretreatment method, saccharification occurs with a small amount of enzymes, so the cost of enzymes can be reduced. Woods with high moisture content immediately after lumbering can be used as raw materials. Since there is no use of chemicals in large amounts, I think there is high potential for commercial bioethanol production at the site of raw material harvesting in the future (such as in Southeast Asia). At the Biomass Technology Research Center where I work, there is a research team that specializes in assessing economic feasibility. We conducted some economic projections at large-scale commercial levels (raw material 1500 ton/day) using the data for pretreatment and saccharification-fermentation processes that we actually conducted in the lab. As a result, there was the possibility for decreasing the cost of bioethanol final product to a level lower than the NEDO's concentrated sulfuric acid method that was considered to have the lowest cost (sufficiently accurate data for economic feasibility are not disclosed for other methods). Since the combination treatment is a multi-step treatment, the cost of the facilities may be higher, but the facility depreciation can be actually the same as the concentrated sulfuric acid method. For example, there is a hydrothermal process in our pretreatment, and the temperature needed is about 150 °C (0.48 MPa) which will not be subject to the strict regulation of the High Pressure Gas Safety

Law (1 MPa or higher), so the maintenance of the plant will be easy. In the future, we shall optimize the process further, and investigate ways to enhance efficiency by using chemicals that is low cost, has low environmental load, and does not affect the following saccharification and fermentation.

## **2 General use of biomass**

### **Question and comment (Koichi Mizuno)**

In this research, using the wood as starting materials, the fermentation process of cellulose and hemicellulose to ethanol through enzymatic saccharification was shown, as well as the ways of manufacturing materials such as polymers from lignin. This may lead to the biomass refinery concept that may replace the conventional refinery using crude oil. What do you think is the effective R&D strategy to promote the biomass refinery?

### **Answer (Takashi Endo)**

Although there is a long history of research for use of lignin,

there has been no definitive technology or product. However, when bioethanol production is commercialized, like in our technology, new lignin different from the one in the conventional papermaking process is produced in large amounts. Looking at the current petrochemical products, crude oil can be used without loss from asphalt and gasoline to plastic. In the future biomass refinery, I think the development of a technology to utilize the wood components without loss and the construction of a social system where they can be utilized are important. To achieve this, I think it is important to create some characteristic products that can only be achieved with biomass products, rather than creating alternatives to petroleum products. To do so, it is necessary to accurately understand the basic properties of cellulose nanofiber and residue lignin obtained from pretreatment, by using analytical methods, and to establish an innovative product realization technology based on the new knowledge obtained and the theories and principles that are already known.

# Restoration of engineering and *Synthesiology*

[Translation from *Synthesiology*, Vol.2, No.4, p.332-337 (2009)]

The Engineering Academy of Japan with its abundant knowledge and experience discusses and issues statements on a variety of topics in engineering. The “Study Group for the Restoration of Engineering” reconsiders the meaning of engineering in the modern world from different perspectives. Since the significance of engineering is closely related to the mission of *Synthesiology*, Akira Ono, Chairman of the Editorial Board of *Synthesiology* interviewed Dr. Kotobu Nagai, a member of the study group, to hear his thoughts on the “restoration of engineering,” and discussed its relationship to *Synthesiology*.

***Synthesiology* Editorial Board**

**Kotobu Nagai: EAJ Study Group for Restoration of Engineering, National Institute for Materials Science**  
**Akira Ono: Editor-in-Chief, *Synthesiology*, AIST**

## Why a study group for “restoration” of engineering?

**(Ono)**

I think the *Kogaku No Kokufuku Kenkyukai* or the “Study Group for Restoration of Engineering” of the Engineering Academy of Japan shares a similar perspective to *Synthesiology*. What is your intention of not using the homonym *kokufuku*, which is a word meaning “to overcome a difficult situation,” and instead using *kokufuku* meaning “to restore the original state of wholeness”?

**(Nagai)**

Recently, there has been talk of a “drift away from science” and “farewell to engineering faculty.” Even though engineering is a very important field, young people are turning away from it, and I’ve always felt that that is a worrying trend. Another point is that the members of EAJ are Japan’s foremost engineers, but they are growing old. All the traditional academic societies of Japan are unable to increase their membership and are aging. It is with a sense of urgency that “something has to be done” that we used the word *kokufuku* meaning succumbing to disease and then regaining health, rather than *kokufuku* meaning to overcome. It is in the sense that we must return to a wholesome state,

that we are intending to start a movement that may promote the value of engineering in such a way that it is appreciated properly by young people.

## Researchers must reconsider the essence of innovation

**(Ono)**

Today, the interest in innovation is high globally, and I think “restoration of engineering” is related to innovation. What do you think is innovation to Japanese industry, society, and people?

**(Nagai)**

While technological breakthroughs are important, in Japan, I think there is a tendency to consider certain breakthroughs as synonyms for innovations.

The people of industry say that the available guiding principles or scientific principles are approaching their limit, and will reach their limits by 2020 to 2030. They know that what they have in hand now has only limited capability to respond to oncoming global changes and new demands and they are seeking ways to stir up real innovation.



Dr. Ono (left) and Dr. Nagai (right)

The rising prices of resources and energy such as that which occurred last year seems to have moved into the background because of the American economic troubles. For society, however, there is an increasing realization that these trends will not change. The way to overcome the social limitations of resources, energy, and the global environment and at the same time create a society where people can live comfortably and securely are the root of the innovations demanded by society.

The fourth phase of the Science and Technology Basic Plan is now starting. The expectations for science and technology have increased and society is making large investments. The technology policy makers are strongly voicing the opinion that the effect of this investment must be made visible to ensure such investments are used to promote industry and the corresponding benefits are returned to the society. This, of course, is a just demand. Yet going back to the level of researchers, they cannot respond to such a demand as long as they believe a “breakthrough is innovation.” The researchers may feel, “I worked so hard to discover or invent this wonderful thing,” but may be unable to fill the gap between the contemporary capabilities of Japanese industry and the state of the art discoveries; society is incapable of utilizing the results. From another perspective, there may be other companies in the world that possess the ability and mindset to use such state-of-the-art discoveries, and the results obtained in Japan may be utilized in other countries, and that would be a problem.

I think the word “innovation” is used to emphasize the fact that we must reconsider how and where a boost can be given to society, going back all the way to the level of individual researchers in order to use the taxpayer’s money for the benefit of Japan.

**(Ono)**

This point is something that I can sympathize with from the standpoint as an editor of *Synthesiology*. I think “going back all the way to the level of individual researchers” is very important.



**Dr. Kotobu Nagai**

**(Nagai)**

The “innovations” talked about by industry and society, and in technological policymaking are different, but there is shared expectation that various breakthroughs will come together into a new direction and give rise to a paradigm shift. For Japan, how do we obtain the necessary resources and energy? We need direction so that we are welcomed and do not become a burden to the world, and we must have firm sense of direction. In that sense, engineering must be at the vanguard, but it is in this sense that it is regretful that we must talk about the “drift away from science” and the “farewell to engineering faculties.”

**Let us define “engineering” as “the science of problem solving”**

**(Ono)**

Regarding Japanese engineering, we had excellent technology but it was lost somewhere along the way. When you say “restoration of engineering,” what do you think is engineering? Do you think there is a science unique to *kogaku* (engineering) that is neither technology nor science?

**(Nagai)**

There is a four-character word *ka gaku gi jutsu*, which is translated “science and technology,” and it is quite difficult to define. I think we must consider *kagaku* (science) and *gijutsu* (technology) separately to seek definitions that are valid today, and then redefine the *kagaku kijutsu*. Some people say “Japanese ambiguity” is good, but ambiguous guiding principles may lead us to failure.

**(Ono)**

First, we must define the words clearly.

**(Nagai)**

*Kogaku* is plainly “engineering” in English. However, some people say *kogaku* is “science and technology and engineering” or “the science of engineering.” Although I understand these points of view, I believe that “*kogaku* is the science of problem solving.”



**Dr. Akira Ono**

However, I am beginning to think that the definition of *kogaku* is beginning to waver in Japan. This may upset some people, but engineering faculties in Japan are approaching something closer to science. There have been comments from people in industry that the meaning of the word *kogaku* has been stretched, and *kogaku* is departing from engineering faculties that are supposed to be the final stronghold of *kogaku*. Recently, many reports blatantly state, “Engineering faculties are no longer pursuing proper engineering but are sub-science faculties or science-faculty-like engineering faculties.” Students who were planning to get into engineering for problem solving decide to go into other areas of science, medicine, or economics. I believe that this happens because engineering faculties are losing their original stance. The concept of engineering faculty was a derivative from the science faculty, and in fact, Japan was the first country that created a faculty of engineering.

### **An age where “science” must evolve alongside “technology”**

#### **(Ono)**

You say that there was an original Japanese *kogaku*. To seek the original definition of *kogaku* is the starting point of the restoration of engineering.

It is said that science and technology are beginning to fuse, while others state, they are mutually exclusive and are evolving separately. What are your thoughts on fusion and exclusivity?

#### **(Nagai)**

The history of Asian science and the history of Asian technology are not studied extensively. While the history of Western technology is not studied heavily in the West, the history of Western science has been extremely well studied. Dr. Yoichiro Murakami, Professor Emeritus of the University of Tokyo, provided a comprehensive explanation: “In the West, there are two things that God created. One is the Bible, and the other is nature. These are the divine revelations. Western science was born because the purpose of scholarship was to clarify divine revelation.”

#### **(Ono)**

One of the origins of Western science was to realize or to seek the divine world according to Christianity. It was closely linked to theology.

#### **(Nagai)**

Yes. On the continent, scientists were supported by royalty, while engineers had relatively low status. In contrast, in England, scientists were considered arrogant and useless, and engineers and craftsmen were favored. In the Renaissance, the encyclopedia was created since “God did not create nature in a systematic manner, but created it randomly and abundantly.

One can approach God if one understands all randomly created things.” It was around this time that the relationship with tools became very apparent in the history of science like, for example, Galileo Galilei needing a telescope to perform his studies or to carry out experiments rather than just thinking.

According to Dr. Murakami, the reason for change in Western science is because the Westerners fought the Muslims. They studied and assimilated the things left behind by the Muslims, and they realized that they failed to properly assimilate Socrates and the Hellenistic culture. A great turning point occurred through their restudying of foreign cultures. This is much like the origin of *Synthesiology*. Some of the theologians decided that things did not have to be systematic, and came up with the encyclopedia style.

#### **(Ono)**

The gathering of facts is foremost in importance.

#### **(Nagai)**

Yes. At around the time of the Industrial Revolution, science and technology ran into each other head on. In technology, making “things” was the objective. Whether it was the printing press, explosives, or a compass—there are theories that the water mill was created in the Orient—, they only had to be constructed and made practical. Principles were unnecessary. I think this is the wonderful part of technology.

Science, on the other hand, is a pursuit of profound truth created by God, and in a sense it does not require results. I think this is wonderful too, and I don’t think either should be regulated in any way. I do think there is an attempt to change from the encyclopedia style.

First, how do we describe the current age? One is that it has become flat, as exemplified by the word “mass globalization.” The world has become very small through the development of traffic and communication. Information can diffuse throughout the world in an instant. It is a new age in this respect.

Around 2050, the world population will be 1.5 times its current value, and about twice the amount of resources and energy will become necessary. The emission of carbon dioxide will at least double. The remaining resources and energy will shrink. After the world population reaches the peak in 2050, the average lifespan will increase. The 21st century is an era when people will age on a global scale.

On the other hand, the speed of development of science and technology will accelerate even more.

#### **(Ono)**

Does that mean that we must accelerate the development of science and technology?

## **Technology backed by science is sought by industry**

### **(Nagai)**

No, I mean that they will accelerate. Looking at the trend of the number of research papers published, the number has increased dramatically with the participation of China and India. As more people participate, new information comes out worldwide at a greater speed. While the probability of serendipity does not change, according to the principle of “more shots more hits,” new concepts will emerge more quickly than before. In this age, anyone who can make use of novelty will win. While there will be chances for scientists and engineers to make big profits, we may end up with terrible consequences for society and humankind. In the sense that science and technology will have to play their significant role with care, a totally new age may arrive.

### **(Ono)**

Does that mean an age where technology and science come closer?

### **(Nagai)**

The speed at which new scientific knowledge and technologies are born and the speed at which they are learned will continue to accelerate. Therefore, unless the technology is excellent and is backed by science from the beginning, it will weaken when adopted by industry. Technology must be supported by science, so I think science and technology must become closer. However, from the standpoint of science, it doesn't really have to approach technology. We do not want to choke science, and I understand very well when Dr. Masatoshi Koshihara says that he'd want to be doing whatever he likes even after several thousand years.

## **Japanese science, technology, and engineering**

### **(Ono)**

I would like to ask about Japanese science, technology, and engineering education. There was a time when Japanese technology was called “copycat technology,” but now Japan is considered to be one of the most successful countries in technological innovation.

### **(Nagai)**

Japanese technology is considered to be extremely advanced by other countries, and I think there are three important aspects to consider. Military technology progress is overwhelmingly dominated by the United States, but not in Japan. So-called civilian technology is regarded hotly by competitors both in Japan and around the world, and as long as Japan and the Japanese possess the capability to handle them, we will continue to create overwhelmingly wonderful things.

### **(Ono)**

I think the fact that the Japanese users are highly quality conscious makes a difference.

### **(Nagai)**

Yes. Second, there is a keen awareness that Japanese products are to be sold around the world. Three, and this is often disregarded, but I speak from the perspective of a metal materials researcher: there is no other country that is so densely populated and experiences serious earthquake damage like Japan, and therefore, the materials used here are the toughest in the world.

As there are several factors that make Japanese technology strong, it has been required to be strong and robust against accidents or disasters, which, in turn, established the Japan brand, and I expect this will continue into the future.

### **(Ono)**

Technologies have developed in their respective regions of the world and have their own logic. In Japan, there is the background of earthquakes, the societal situation of low birth rate and aging, and issues regarding energy. These factors provide mechanisms that nurture the growth of technology, and with a proper mindset, it has worked very well so far.

While it has excellent technology, how about Japanese science?

### **(Nagai)**

It is quite difficult to respond in a simple way to the question, “why do we do science?” In terms of matching up to first-class science in the world overall, Japan does not hold a good position. At this point, we are number one in the field of materials, but China is close behind us.

We have entered an age of mass globalization, and all information diffuses widely. However, when seeking optimal solution, localization becomes very important. We cannot solve a problem if we are simply copying other countries. This is true for science. However if we say, “Don't copy other countries,” people won't copy at all, but we must copy certain things. There must be diversity. I think if we look back at the excellence of Japanese technology, we can find its origins in various new topics of research.

### **(Ono)**

For Japanese technology, I can describe characteristics that make it excellent, but I cannot find any for science. At this point, the only good point I can see is “we don't copy,” and that is a harsh reality.

There was a time when Japanese engineering shined.

**(Nagai)**

In our study group, we analyzed what were the greatest achievements in Japanese engineering after WWII. We can say with confidence that it is the factory system. The supply chain, and everything from control, management, and operation technology in total is absolutely wonderful. Seen from the world perspective, Japanese plant technology is the textbook of textbooks.

**(Ono)**

Do you mean it's not only about introducing industrial robots?

**(Nagai)**

On the other side, there is no hesitation in introducing industrial robots, and there has been the wisdom and tradition to do so. While the layoff of laborers by introducing industrial robots must be considered a separate issue, technological innovation can be extremely severe and inevitably results in sacrifices. I don't think technological innovation will make all people happy, and there will always be people who suffer loss. Wisdom is required to see the correct direction while weighing the welfare of society in total.

**Engineering education is not studying "science" but studying "what is science"**

**(Ono)**

Regarding Japanese engineering education, you mentioned today we are saying "farewell to engineering faculty," and that's unfortunate. What made the engineering faculty shine?

**(Nagai)**

I said Japan was the first country in the world to create an engineering faculty. At the beginning of the Meiji Period, the Ministry of Engineering created the Imperial College of Engineering, and the world's first engineering faculty was established. Henry Dyer, a Scotsman, was invited to Japan to design the education plan. At the time, the ministries had college-like institutions. It was like the Ministry of Economy, Trade and Industry running a university.

I read Henry Dyer's "Valedictory Address to the Students" at the first graduation ceremony, available at the library of the Tokyo Institute of Technology. It was written: "On the continent, the status of science is high and technology is placed below it, and good results could not be obtained. In England, technology is given an extremely high status, but it is unrelated to science, and although the results are good, they engage in trial-and-error indefinitely so development cost is high. I was invited to Japan because I wanted to design new education paradigm by taking the best of both." To carry out a new type of university education for the transmission of skills and for teaching world's most advanced science, the

practicum and lectures were very well combined. Of the six years in school, the 5th and 6th year students went to the site of the Ministry of Engineering to do hands-on work, and were required to write a graduation thesis.

The address includes points such as: the university must properly manage the library and resource rooms so the students can go and study the best works of their forefathers; an academic society must be established; or one must study foreign languages to directly learn state-of-the-art knowledge. These are common sense today. It is also written that one must actively study literature and religion. To think why one is studying cannot be done without such knowledge. He goes on in the lecture that "when you start to work and meet people from other countries, whether you can name one literary work of that country will affect how you will be received."

**(Ono)**

I hear that story now, but I see it started back then.

**(Nagai)**

He talked of ideals, but I think the ideals ring true to this day. As criticism of the continent, he already said, "In engineering, one is less able to make anything good when he is packed with more knowledge. We must have the students study 'what is science' and there is no need of studying 'science.'" I understand this thoroughly.

**What "engineering" can do in the 21st century, the age of collaboration and competition**

**(Ono)**

Did Japanese engineering shine for the second time in "monozukuri" or "manufacturing" centering on industrial technology after the WWII?

**(Nagai)**

I think it is "monozukuri" or "manufacturing" itself. When it is called "transmission of skills," it invokes an extremely personal image, and I don't find it useful. However, if a skill is transmitted without the spirit of manufacturing, people may say, "We don't want it." This is written in "To Those Aspiring to Be Scientists" used by the National Academy of Engineering (USA), and has been mentioned by Henry Dyer, but in my own words, I think human beings are the only animals that can have dreams or have curiosity in that they wish they had some thing, and can use the materials in the environment to create and realize those dreams. Curiosity is born when one comes across different things or different thinking styles that he/she never imagined existed. In my generation, my curiosity was raised by seeing things that did not exist in the real world in the manga "Atom Boy." To come into contact with different things, and to actually try them out— if it works, you may be convinced that you are

a genius, but that is important. Then, you must go out to the world and challenge established ideas. I think that is what “the age of collaboration and competition” means.

In an age where a problem cannot be solved with only one idea, to remove the walls and to share knowledge and ability are something that only humans can do. Of course, the basics are important. While merely packing in knowledge won't work, education is important.

**(Ono)**

What you said is certainly the spirit of *Synthesiology*. I think this will lead to the third success of engineering in Japan.

**(Nagai)**

From the standpoint of *Synthesiology*, I think it is necessary to build such mechanisms and activities at all levels and in all places. The key point is education in high school and above. It is necessary to create an environment where different ideas can compete. This means to nurture “dreams” and “curiosity.”

### **Design is the essential human ability and the origin of engineering**

**(Ono)**

Is this related to “design is the essential human ability”?

**(Nagai)**

I mentioned it for that purpose. Taking it to an extreme, I believe we can go ahead and create a new engineering faculty by combining the current faculties of economics, law, and engineering of Japan.

**(Ono)**

The general term for all of that will be design.

In our discussion today, your contrast of science and technology was very interesting. While Western science has its origins in the clear objective of seeking God's design, there is no clear explanation of why science exists in Japan, and it's not going to endure by just being interesting. However, Japanese engineering had shone bright twice so far, and it is trying for its third glory, and I see that it is in good standing. This must be stated clearly, and the flag must be flown visibly. This is perhaps the same as strategy making.

**(Nagai)**

I think strategy making is important. It will be an issue for the third shining age of engineering.

**(Ono)**

Thank you very much for a valuable discussion.

(This interview was conducted at the National Institute for Materials Science in Tsukuba on August 19, 2009.)

### **Profile of Dr. Kotobu Nagai**

Completed the masters program at the Faculty of Engineering, The University of Tokyo in 1977. After working as assistant at the Faculty of Engineering, UT, transferred to the National Research Institute of Metals (which became the National Institute for Material Science in 2001) in 1981. Worked as the head of Mechanical Engineering Lab, head of the Steel Research Center among others. Currently the Area Coordinator in charge of the Environment and Energy Materials. Doctor of Engineering (The University of Tokyo 1981). Member of Science Council of Japan. Specialty is metal material science (basic themes are clarification of the relationship between microscopic structure and mechanical properties; and compatible design of low environment load and high performance).

# Editorial Policy

*Synthesiology* Editorial Board

## Objective of the journal

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

## Content of paper

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

are related to each other, what are the problems that must be resolved in the integration process, and how they are solved (Item 5). Finally, there should be descriptions of how closely the goals are achieved by the products and the results obtained in research and development, and what subjects are left to be accomplished in the future (Item 6).

## Subject of research and development

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

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

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

## Peer review

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

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

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

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

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

## References

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

## Types of articles published

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

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

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

# Instructions for Authors

*Synthesiology* Editorial Board  
 Established December 26, 2007  
 Revised June 18, 2008  
 Revised October 24, 2008  
 Revised March 23, 2009

## 1 Types of contributions

Research papers or editorials and manuscripts to the “Readers’ Forum” should be submitted to the Editorial Board.

## 2 Qualification of contributors

There are no limitations regarding author affiliation or discipline as long as the content of the submitted article meets the editorial policy of *Synthesiology*, 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 editorials should also comply with the same structure and format except subtitles and abstracts are unnecessary. Manuscripts for “Readers’ Forum” shall be comments on or impressions of articles in *Synthesiology*, or beneficial information for the readers, and should be written in a free style of no more than 1,200 words. Editorials and manuscripts for “Readers’ Forum” will be reviewed by the Editorial Board prior to being approved for publication.

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 and editorials shall have front covers and the category of the articles (research paper or editorial) 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 with names of reviewers disclosed, and the Editorial Board will edit the highlights of the review process to about 3,000 Japanese characters (1,200 English words) or a maximum of 2 pages. The edited discussion will be attached to the main body of the paper as part of the article.

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.

3.3.2 The text should be in formal style. 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, clear originals that can be used for printing or image files (resolution 350 dpi or higher) should be submitted. In principle, the final print will be 15 cm × 15 cm or smaller, in black and white.

3.3.5 For photographs, clear prints (color accepted) or image files should be submitted. Image files should specify file types: tiff, jpeg, pdf, etc. explicitly (resolution 350 dpi or higher). In principle, the final print will be 7.2 cm × 7.2 cm or smaller, 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).

## 4 Submission

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

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c/o Publication Office, Public Relations Department,  
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E-mail: [synthesiology@m.aist.go.jp](mailto:synthesiology@m.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 are allowed in the proofreading stage.

## 6 Responsibility

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

## 7 Copyright

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## Letter from the editor

*Synthesiology* Volume 2, Issue 4 has been published. This issue contains diverse research papers just like the previous issues. One of the characteristics of *Synthesiology* is that the reader can gain some kind of insight upon reading any of the papers. I shall mention three points that I felt strongly.

First, reading the papers, I realized I could not immediately pinpoint to what discipline a paper belonged. Although it may be possible to determine the applicable discipline from the institution where the author works, his/her resume, or his/her subject of study, however, that would be somewhat meaningless. That is because the act of combining multiple technological factors for a certain purpose is an act of transcending the framework of a traditional field, and some research cannot be categorized by traditional disciplines. Then, I realized if I could not clearly discern the discipline of a paper, it must be research conducted in true synthesiology style.

Second, as I have already written in the “Letter from the Editor” of the previous issue, the papers in *Synthesiology* are wildly varied compared to the academic journals that are published by disciplines. I mentioned the stakeholders last time, and this time, I see words such as price, uncertainty, cyclic development, real time, and social use. Such terminologies are seldom mentioned in conventional academic journals. In general,

analytical researches place importance on discoveries and champion data, whereas researches in *Synthesiology* seek social utility. This leads to the differences in the points of emphasis and the ways papers are written. The facts that the discipline cannot be pinpointed and that there are added perspectives and issues unseen before are indications that the researchers who submit the papers and the reviewers who review the papers are beginning to understand the objectives of *Synthesiology*.

Finally, although currently a majority of the papers are submitted by the researchers of AIST, I expect more submissions from industry and academia. The Editorial Board is engaging in discussion everyday on how the editorial system should be and what we can do to increase the participation particularly from the people of industry. Also, while this may be extremely ambitious, alongside the practical papers that aim at synthesis of technology, I am expecting a paper that attempts to establish a synthesis methodology by studying the papers of *Synthesiology*. If our aim is to establish synthesiology as a new science of synthesis, we must make attempts at generalization of its methodology.

Editor in charge of Partnership  
Koh Naito

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K.Ohba

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*-An advanced mechanical design for the iodine stabilized He-Ne laser-*

J.Ishikawa

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