

Novel Passive Optical Fuse Created by Inserting Carbon-coated Low-Melting Glass in Optical Fiber Circuit

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With recent progress in high-output laser sources, systems which handle more intense light have come into widespread use. One example is multiple wavelength transmissions, in which 200 or more optical signals with different wavelengths are transmitted over a single optical fiber. Under these conditions, some type of protective measure is required, as optical components of the system can fail if exposed to an excessive incident beam.

Incorporation of an optical fuse in optical systems, analogous to the fuses used in most electrical products, is the most direct solution. Realistically, however, optical transmission media do not display any phenomenon which corresponds to the Joule heat (proportional to the square of current) which is used to activate electrical fuses. Conventional optical systems must therefore employ devices which monitor the intensity of the incident light and break the circuit when necessary, but these devices require a separate power source.

The Functional Glass Group discovered a phenomenon which can be used in an optical fuse with no external power source. This can be observed in a structure consisting of an optical fiber circuit inserted by a low-melting glass (a general example of this structure is shown in **Fig. 1**), which is coated with a carbon-containing material. The appearance of the device is shown in **Fig. 2(1)**. The carbon coating material, which appears as black in the photo, contains silica glass single-mode optical fibers (diameter: 0.125 mm) for use in optical transmissions, which are arranged with the ends facing each, and the low-melting glass, which connects the fibers. Virtually all of the light moving through the optical fiber passes through the transparent glass and arrives at the second optical fiber, but a very small remaining amount of light reaches the carbon material.

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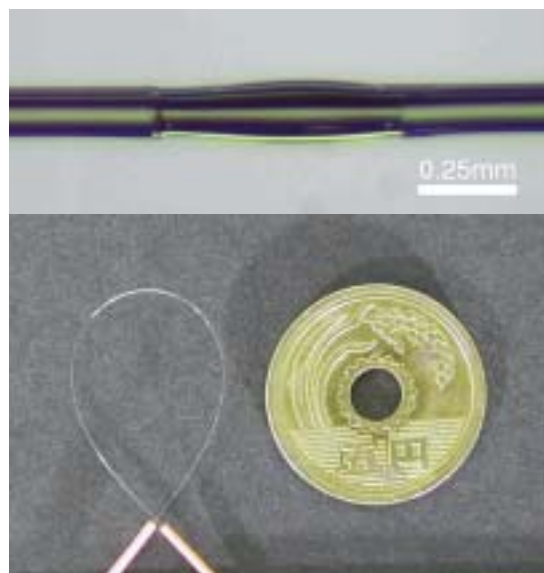


Fig. 1 Optical coupling structure between two ends of silica glass optical fibers by inserting a low-melting glass. This structure can withstand this degree of bending. (Lower right: A 5-yen coin with the diameter of 22 mm).

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High Durable SiAlON Phosphors

- The Best Suited Materials for White LEDs and Next-generation Displays -

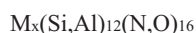
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High durability is generally required for those phosphors applied in the next-generation lighting and displays such as white LEDs (light-emitting diodes), PDPs (plasma display panels), and FEDs (field emission displays), as they are usually exposed to excitation under high brightness light for long periods.

Currently available phosphors are usually based on oxidic or sulfidic host lattices, which are activated by rare-earth ions. However, a serious problem arises from the decomposition of the materials in service and thus the brightness degradation over time due to their weak crystal structure. To solve this problem, the Non-Oxide Ceramics Group has proposed -SiAlON , a well-known heat-resistant material, as a new host material for phosphors. We have

found that the -SiAlON phosphor possesses excellent photoluminescence properties, in combination with high thermal and chemical stabilities.

-SiAlON is a solid solution of silicon nitride which has a general chemical formula as below:



Where, M is a solid solution metal. Phosphors can be realized by using Eu or other optically-active rare-earth ions as the solid solution element. These new phosphors have the following advantages over conventional oxide phosphors.

High durability: Due to the high thermal stability of the host lattice, the brightness degradation is limited, and changes in photoluminescence efficiency and color over time are minimal.

Visible light excitation: Most oxide phosphors can be only excited by UV light. The -SiAlON phosphor, however, promises the excitation under visible light as nitrogen is introduced in the crystal structure.

Better flexibility in material design: It is allowed to synthesize materials with varying M and the N/O ratio, and thereby to modify the spectrum suitable for intended applications (**Fig. 1**).

These novel phosphors offer the promise for applications in white LEDs and the next-generation displays. White LEDs can be produced by coupling a blue LED to a yellow phosphor, generating white light based on the complementary color relationship between blue and yellow. To this end, a yellow phosphor which can be excited under visible light with a wavelength of 460 nm is essential.

As shown in **Fig. 2**, Eu- -SiAlON has an excitation peak at around 450 nm, and thus emits light with good efficiency when excited by a blue LED. It is yellow in color with the emission peak at 550-590 nm, allowing it to be a good phosphor for white LEDs with appropriate luminescence properties. Moreover, these phosphor materials are also well-suited to the applications in displays which are required a service life of several 10,000 hours.

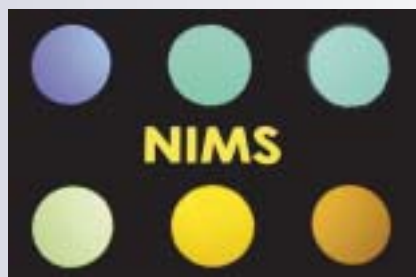


Fig. 1 Multicolored phosphors by appropriate material design.

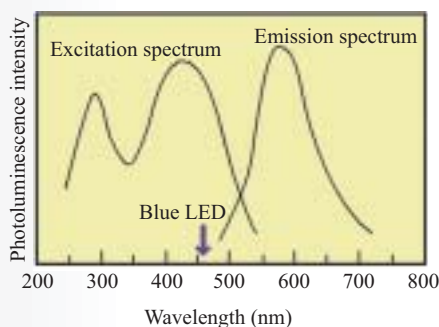


Fig. 2 Excitation and emission spectra of Eu-doped -SiAlON .

Development of Painless Hepatic Function Test Chip

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With present techniques, hepatic function tests for alcoholic liver damage and hepatitis require from several hours to several weeks from blood sampling until test results are available, and, moreover, blood samples must be taken by qualified professionals. For both reasons, the development of a convenient device which allows immediate testing during medical exams and enables home use without special training has been desired.

To meet this need, our group and a leading semiconductor maker, Rohm Co., jointly developed a chip which makes it possible to test hepatic function at home by taking blood samples with a painless needle mechanism, and also established the basic technology for this method.

For pain-free blood sampling, multiple needles 0.5 mm in height are fabricated on a silicon chip, as shown in **Fig. 1(a)**. The needles are inserted into the skin by a spring mechanism (**Fig. 1(b)**), and a minute blood sample is taken from capillaries. (The sample would fill a 2 x 2 mm cube.) The patient feels virtually no pain. The blood sample is then extracted through numerous holes in the silicon chip, led to a ultra-small channel connected to the chip, and separated into blood cells

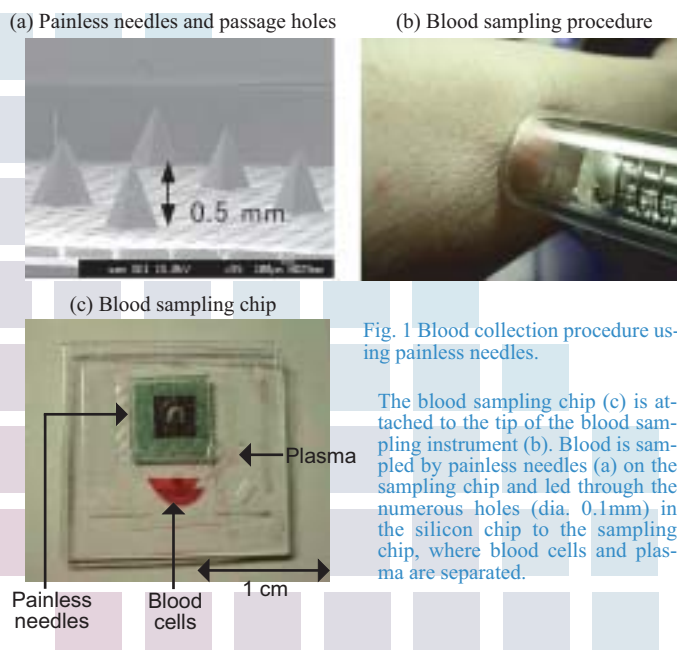


Fig. 1 Blood collection procedure using painless needles.

The blood sampling chip (c) is attached to the tip of the blood sampling instrument (b). Blood is sampled by painless needles (a) on the sampling chip and led through the numerous holes (dia. 0.1mm) in the silicon chip to the sampling chip, where blood cells and plasma are separated.

Non-linear Lattice Relaxation and Light-Emission Process

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- Dynamics of Lattice Relaxation Process in Polymers -

In functional polymers, represented by polyacetylene, electron systems enclosed by 1-dimensional nano-spaces on the main chain skeleton of the polymer cause characteristic optical functions, which include large non-linear optical effects, exciton stabilization, and photo-induced structure changes.

In polymers, photoinduced excited states cause deformation of the surrounding lattice, giving rise to various kinds of lattice relaxed states, including light emitting self-trapped excitons, polarons, either neutral or charged solitons, which are "kink" of the main chain, etc. Because the formation and competition behavior of these lattice relaxation states is intimately related to the occurrence of optical functions, a clarification of the dynamics of the initial formation process would be particularly important for elucidating the mechanism optical function and creating high functions.

For a detailed investigation of the optical properties of polymers, experiments were conducted with single crystals of organic-inorganic hybrid polymers (halogen bridge mixed valence metal complexes) with physical parameters including electron correlation, electron transfer energy, electron-lattice interaction, etc. varied over a wide range. Concretely, the initial lattice re-

laxation process was investigated by measuring changes over time in the absorption spectra after irradiation with 100 femtoseconds time resolution.

When electron - hole bound states (excitons) are photo-generated, relaxation processes of vibronic excited self trapped excitons to quasi-stable states releasing excess (thermalization) was observed as a shift in the photo-induced absorption band. On the other hand, a formation process of neutral solitons, which compete with self-trapped excitons in the formation process, within the time-resolution was found to exist (Fig. 1). When free electron - hole pairs are pho-

to-generated, a process of polaron stabilization with associated vibration was observed. In this case, the time constant for thermalization of the vibronic excitation state was 1.1 picoseconds (Fig. 2).

Future directions for study include investigation and clarification of phenomena such as changes in the dynamics of lattice relaxation, group phenomena, and photoinduced phase transition, etc. with a wide range of changes in the physical parameters which control physical properties, as a theoretical basis for developing higher functions and new applications for high polymers.

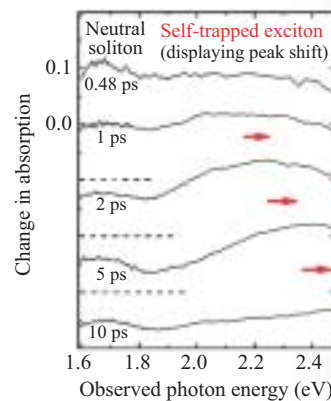


Fig. 1 Time-resolved photoinduced absorption spectra of self-trapped excitons and neutral solitons in Pt-Cl complex.

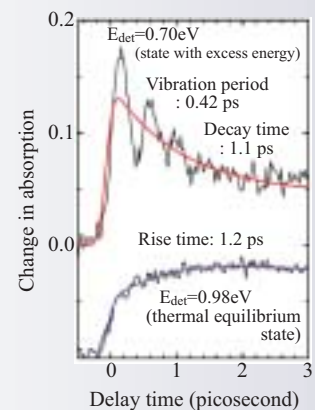


Fig. 2 Time evolution of photoinduced absorption intensity due to thermalization of vibronic excited states of polarons in Pt-I complex.

and plasma by a centrifugal separator (Fig. 1(c)). The plasma is mixed with a test reagent by a micro-mixer and led to the measurement channel, where it is irradiated with light and analyzed by the color test method. Results can be obtained in about 5 minutes, allowing doctors to perform this test as part of examinations.

The hepatic function test chip which was test-manufactured in this project (shown in Fig. 2) tests three types of liver function, -GTP, GOT, and GPT. The chip substrate is made from the same plastic as PET bottles and is inexpensive and disposable.

As an urgent near-term task, we are now working to complete a single chip which performs all functions from blood sampling to measurement, as shown in Fig. 3. After improvement in measurement accuracy and confirmation of stability, applications will be filed for approval as a medical instrument.

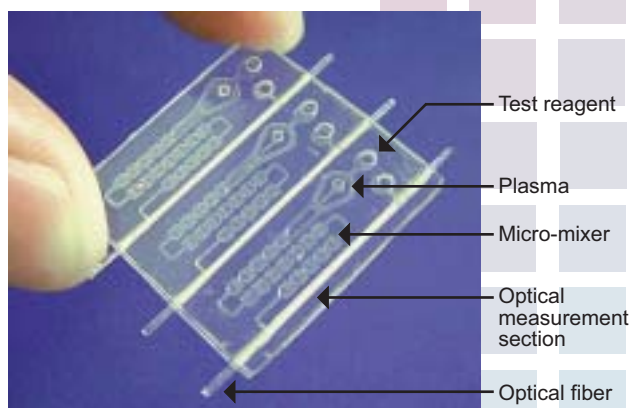


Fig. 2 Hepatic function test chip for three markers of liver function.

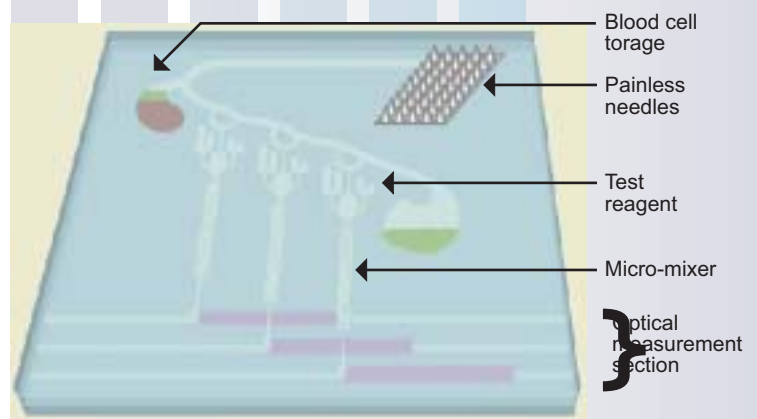


Fig. 3 Integrated colorimetric analysis chip for hepatic function examination which is under development.

UV Light Emission in cBN Single Crystal with p-n Domain

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In recent years, the development of short-wavelength solid-state light-emitting devices has become an important research topic in IT-related fields. However, in order to generate short-wavelength emissions, it is necessary to fabricate a p-n junction in a semiconductor material with a wide band gap.

Cubic boron nitride (cBN) appears to be an excellent material for this purpose, as it has the largest band gap among compound semiconductors known to date. Consisting of boron and nitrogen, cBN is the simplest III-V family compound. It is an artificial substance which does not exist in nature, and it has a crystal structure similar to that of diamond. Reports have mentioned synthesis of p-type and n-type crystals of cBN, and UV light emissions resulting from the p-n domain between the two, but due to quality problems with the single crystals obtained thus far, as well as poor yield in fabrication of light-emitting devices, little progress had been made in understanding their properties.

The High Pressure Group therefore carried out research on high pressure synthesis of high quality cBN single crystals and clarified their basic semiconductor properties. Among other findings, in crystal growth under high pressure, we discovered that (1) cBN shows an n-type semiconductor property due to imperfections introduced during synthesis, and (2) beryllium (Be), which is added to obtain a p-type crystal, accumulates in a designated growth orientation within the crystal ((111) boron growth sector,

etc.). This means that a p-type domain of accumulated Be can be formed within an n-type semiconductor single crystal by properly controlling the concentration of the Be acceptor, and a p-n bond interface (transition region) will form spontaneously within the crystal.

Fig. 1 shows light emissions when a voltage is impressed from the p-n boundary (transition region) formed spontaneously within the single crystal. As shown in **Fig. 2**, important light-emission properties included a broad intensity distribution with a peak at around 300 nm.

To date, light emissions corresponding to the band gap of cBN (wavelengths up to approximately 200 nm) have not been obtained, but this is considered to be a result of imperfections inherent in the crystal. We therefore produced higher purity cBN crystals and found that free exciton light emissions (light emissions corresponding to the band gap of the high purity crystal) occur under electron beam irradiation (cathode luminescence).

Future goals include the fabrication of unique devices which display deep-UV light emission from the self-forming p-n domain in single crystals based on further progress in impurity control.



Fig. 1 Light emissions from cBN single crystal with spontaneously-formed p-n domain. (Left) before impressing voltage, (right) after impressing voltage.

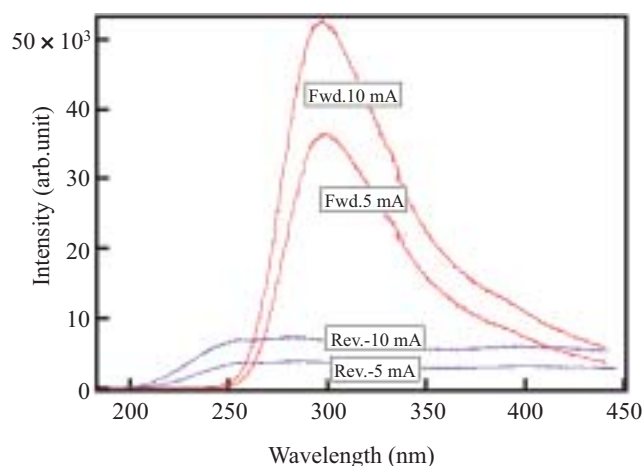


Fig. 2 Light-emission spectrum of single crystal.

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Novel Passive Optical Fuse Created by Inserting Carbon-coated Low-Melting Glass in Optical Fiber Circuit

When the intensity of continuous wave light (wavelength: 1.54 μm) passing through this optical fiber circuit was increased, a sudden flash occurred at around 1.3 W, as shown in **Fig. 2(2)**. From the **photo** in **Fig. 2(3)**, taken immediately after the flash, the low-melting glass has become deformed and the connection between the two optical fibers is broken. The cause of this flash is thought to be intense heat generated instantaneously by the reaction to the excessive incident light reaching the carbon coating. This heat selectively deforms the low-melting point glass, breaking the optical circuit.

In the future, we plan to clarify the controlling factors in fuse behavior by investigating materials and device configuration.

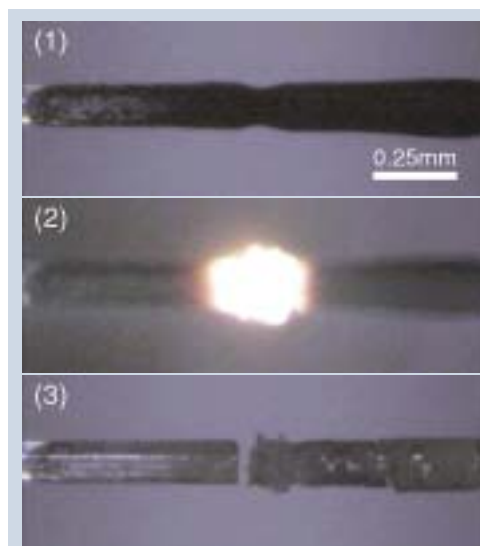


Fig. 2 Circuit-breaking phenomenon triggered by excessive incident light.

For further information, please visit: http://www.nims.go.jp/glass-lab/index_e.html

Control of Fluorescent Characteristics by Modification of Interlayer Interface

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Clay minerals and other inorganic materials with a layered structure are known to incorporate cationic organic molecules between their layers (intercalation: **Fig. 1**) to form a sandwich-like 2-dimensional layered nano-composite. The existence of these substances suggests that functional organic dyes such as laser dyes could be incorporated in this type of structure, as a kind of synthetic mica, and applied to solid-state dye lasers.

In theory, it should be possible to increase fluorescent intensity by increasing the concentration of the laser dye. However, in practice, organic dye molecules tend to coalesce and form aggregates at high concentrations. In particular, fluorescent emission, which is the source of the laser emission, vanishes in H aggregates, where the molecules are arranged in parallel rows.

As one example, when the laser dye rhodamine 6G is introduced between the layers of a clay mineral (layered silicate compound such as montmorillonite), an H aggregate forms and fluorescence is no longer observed. This is because the interlayer space in clays is hydrophilic and shows little affinity (organophilic) for organic substances. As a result, the dye molecules tended to aggregate with other molecules of the same kind. To prevent this, it is necessary to modify the interlayer space to an organophilic environment which encourages dispersion of organic molecules. Based on this thinking, an organophilic surfactant with a long alkyl-chain group was incorporated in the interlayer space in advance, forming an organophilic mica, and rhodamine 6G was then introduced into the interlayer spaces, producing a composite with strong fluorescence.

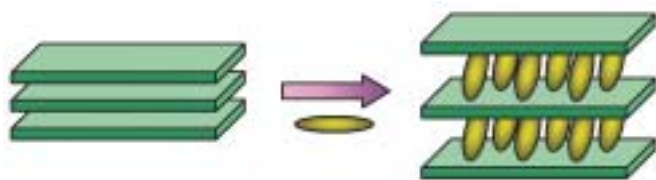


Fig. 1 Concept of inclusion of organic dye molecules in 2-dimensional clay interlayer space. Green shows inorganic mica layers; yellow shows laser dye molecules.

Thus, the interlayer environment (hydrophilic-organophilic, etc.) greatly influences aggregate formation and fluorescent properties. Because these properties would be controlled by organophilic-mismatches, fluorescent emission properties were investigated with pyrromethene, a laser dye similar to rhodamine 6G, using organophilic micas synthesized with various surfactants. The results showed that organophilic mica containing a surfactant with one chain such as cetyl trimethyl ammonium displays stronger fluorescence, by one order of magnitude or more at maximum light intensity, than mica with a surfactant having two long alkyl-chain groups (**Fig. 2**).

Although complete control is not yet possible, we are attempting to achieve higher level control of the arrangement and interactions of organic molecules in these nano-composites, with the goal of applying this class of substances in solid-state dye lasers and similar. As a further stage, we are also studying extension of this technology to materials which actively utilize molecular orientation, by realizing a regular arrangement of a large number of molecules in the 2-dimensional interlayer space.

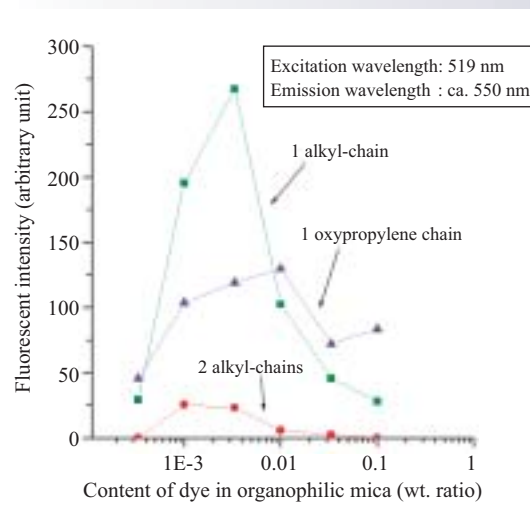


Fig. 2 Fluorescent intensity of laser dye (pyrromethene) in interlayer space of organophilic micas incorporating different surfactants.

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Second NIMS - NEERI Workshop on "Emerging Materials for Environmental Applications"

The NEERI-NIMS joint workshop on "Emerging Materials for Environmental Applications" was organized at NEERI (Nagpur, India) from January 20 to 21, 2004 under NIMS/AML-NEERI MOU concluded in 2002. Four researchers headed by Dr. H. Haneda (Director, ECG) participated from NIMS. The opening talk of Dr. S. Devotta (Director, NEERI) was followed by the 18 scientific presentations. This workshop was focused on recent advances of green chemistry, various environmental materials and applications, and also on direct interactions not only between NEERI and NIMS scientists but also to include scientists from other Indian R&D institutes. After the plenary lecture and scientific presentations, a panel discussion was organized to summarize and to identify potential areas for collaborative research. It was resolved to make various efforts for aiming at further development of our collaboration works.

Research Exchange Meeting with Central Research Institute of Electric Power Industry (CRIEPI)

On January 20, NIMS and CRIEPI held a regularly-scheduled research exchange meeting at CRIEPI's Head Office in Otemachi, Tokyo, with participation by a total of 40 members of two organizations. The meeting included an introduction of recent research topics and exchange of ideas on tasks and strategies for promoting cooperation.

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