Hitachi Chemical

Working On Wonders

# Hitachi Chemical Technical Report

No.57 ／ March 2015


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## Review



## Report




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# COMMENTARY

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# Electrical Energy Storage Business of Hitachi Chemical Contributing to the Secure Social System and Environmental Conservation

Nowadays, mid- to long-term issues which significantly impact on social environments, such as global warming, natural resource problems and the advance of an aging society, are piled up. At Hitachi Chemical, we have defined "Information/Communication & Display", "Automobile & Transportation infrastructure", "Environment & Energy" and "Life Science" as our priority business areas and are working to create new products and new businesses as well as offering various materials and components. Specifically we aspire to expand our business in the focal areas of "Environment & Energy" and "Life Science", where significant growth is set to be achieved.

In the environment & energy business sector, Shin-Kobe Electric Machinery Co., Ltd. became our wholly owned subsidiary in April 2012 to help catalyze our core electrical energy storage device business, swiftly respond to facilitate globalization of our business and consolidate our energy storage business. Thanks to this merger, new product development is being accelerated, amid positive synergy by exploiting the competitive advantages of battery development/manufacturing and power supply system technologies owned by Shin-Kobe Electric Machinery and material technology together with material analysis and interpretation skills which we have nurtured since our company was founded.

At Hitachi Chemical, we are expanding our electrical energy storage device business in two areas, namely industrial and automotive applications. In the industrial area, people have become acutely aware of the need to secure power supply sources in emergencies or the tight supply-demand balance and save electrical power and energy by using electrical power more efficiently in the wake of the Great East Japan Earthquake. Moreover, business enterprises are also promoting such measures from the business continuity planning (BCP) perspective. The market for electrical energy storage systems is henceforth expected to grow as the key equipments, to shift peak load and energy management in targeted areas and communities during normal operation as well as emergency electrical power sources. In addition, amid efforts to counter global warming, there are rising expectations of non $CO_2$-emitting renewable power sources, such as wind power and solar power (PV). However, the issue of power system instability emerges if a considerable proportion of electricity is generated from renewable energy sources. This is due to irregular fluctuations in electric power generation, which is heavily dependent on weather conditions, in response to which the increased integration of electrical energy storage systems is expected to mitigate and stabilize output power fluctuations. Our company has already developed and launched an electrical energy storage system using a lead-acid battery with an expected service life of seventeen years and started delivering the same since 2009. In the industrial sector, our company has continued to promote the lead-acid battery business mainly including backup power systems for office equipments

and phone base stations and the capacitor businesses for power inverter circuits of wind power/solar power (PV) generators and various power supply sources. We are determined to meet needs appropriately as mentioned above and focus on developing and commercializing electrical energy storage devices and systems, which can contribute to a steady energy supply and help achieve a low-carbon society while continuously developing a new device business such as lithium-ion batteries and capacitors. In the automobile sector, development and popularization of fuel-efficient cars, are advancing especially cars with idling stop systems (ISS) are expected to become particularly popular, because of the fuel efficiency can be improved economically. Our company continues to focus on improving the performance of lead-acid batteries for cars with ISS and help reduce carbon dioxide emissions by boosting the fuel efficiency of cars with ISS.

In response to our prevailing business environment, we strive to grow our electrical energy storage device business into the third core business following those of high-performance materials and automotive parts. One great feature of our business portfolio is possessing the four distinctive electrical energy storage device products, namely lead-acid battery, lithium-ion battery, lithium-ion capacitor and conventional capacitor businesses and further power supply equipments and electrical energy storage system products. We strive to continue growing our device business by optimally exploiting our advantage, with hybridization as one option. Various electrical energy storage system needs are required, depending on the applicable capacity zone and applications, and meeting such needs with a single type of device is not always appropriate. In this respect, our hybrid electrical energy storage system, which combines multiple types of unique devices, can optimally fulfil wide-ranging requirements in terms of performance, cost and other needs. We want to provide electrical energy storage solutions optimized for various applications by enhancing the features of four different electrical energy storage devices and combining their performances. We will also continue further globalization and expansion of our business portfolio. We accelerate expansion of our business cooperatively as a team with Hitachi Group companies and by utilizing the abundant resources available in the Hitachi Group. As for global deployment, one example includes the power grid stabilization business in North America, where we started verification tests on an ancillary (frequency regulation) service in June 2014, using a container-type electrical energy storage system “CrystEna: developed by Hitachi, Ltd.” incorporating our lithium-ion batteries. As for expansion of our business portfolio, we attempt to expand our business into new electrical energy storage system applications as the main focus, also including battery status monitoring and service businesses such as energy management and maintenance.

Our company is developing a range of products, in environment & energy business fields including high-performance materials for solar cells and other materials for wind power generator and thermal management as well as electrical energy storage devices. In this Hitachi Chemical Technical Report, we would like to introduce some of our activities in environment & energy fields. As the environmental & energy issues becoming more serious, we hope to play our part in solving and overcoming these issues and achieving a low-carbon society. We hereby pledge to follow through with our corporate philosophy, which involves contributing to society through technical advancement and products that can herald a new era, toward the realization of a sustainable society.

# Electrical Energy Storage Devices & Systems

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Expectations of renewable energy as a measure to solve the issues of global warming and energy resource shortage are increasing. However, concerns over the unstabilization of power grids are growing, as the amount of wind power and photovoltaic energy sources—the generating power of which fluctuates intensely—increases, and stabilizing the electrical power grid is becoming important issue. On the other hand, experiences of long-term blackouts and following electricity shortages and restrictions after the Great East Japan Earthquake have increased awareness of the need for power source decentralization and high-level energy management for further stable electric power supply and energy conservation. Electrical energy storage systems are expected to increase their important roles as key facilities to solve the above issues. In this paper, products and technologies of four electrical energy storage devices & systems contributing to energy storage systems and energy conservation of various types of energy managing equipment are overviewed.

## 1 Introduction

In recent years, more and more phenomena have seen sparking grave concerns over influences due to the steady progress of global warming, such as frequent abnormal weather worldwide. As an ace card to counter global warming, the widespread use of renewable energy sources is expected, such as wind and solar power. It is also becoming more important to utilize renewable energy from the viewpoint of energy security, given the increasing scarcity of fossil fuels, which is set to be exacerbated by growing demand for energy resources following economic growth in developing countries. Although many countries have been promoting the introduction of renewable energies, concerns are rising over the unstabilization of power grids caused by increased wind power and photovoltaic (PV) energy sources—the generating power of which fluctuates intensely depending on weather conditions. As a countermeasure, the importance of mitigating fluctuations in output power and stabilizing power grids by introducing electrical energy storage systems is becoming prominent.

On the other hand, experiences of long-term blackouts and following electricity shortages and restrictions after the Great East Japan Earthquake have increased the interest in having a stable power supply and boosted awareness of the needs for power source decentralization, secure energy supply near demand area, and high-level energy management, particularly electric power for further energy conservation; not only during emergencies but also under normal circumstance. Various efforts to improve over all energy efficiency by introducing high-level energy management systems into a range of areas, such as house, building, factory, commercial facility or community, and networking power using equipments with electrical energy storage systems are already underway. The market for electrical energy storage systems is expected to further proliferate, not only as emergency power sources but also key facilities of energy management.

Continuing actions to conserve energy and reduce carbon dioxide emissions have further intensified. Since automobiles are considered a major carbon dioxide emission source, a target of around 30% additional reduction of fuel consumption regulation value for 2020 compared to the 2014 figure is under consideration for developed countries, including Europe, Japan and North America[1)], while the introduction of fuel economy standards comparable to those in developed countries is also being considered for developing countries such as China. Various electrification systems and systems to enhance power-train efficiency for better fuel efficiency are also predicted in future.

To realize a low-carbon society, a comprehensive approach will be required encompassing expanding the use of renewable energy and boosting the efficiency of existing facilities on the supply side, and striving to conserve energy from both hardware/software perspectives on the demand side, i.e. not only technological energy saving for devices and systems, but also savings in electric power and energy by changing consumer behavior. In this paper, our electrical energy storage devices/system products which help save energy in hardware perspective and technologies to support these products are introduced.

## 2 Possessed Technologies and Products

In April 2012, the Hitachi Chemical group took over Shin-Kobe Electric Machinery Co., Ltd. as a wholly owned subsidiary to further enhance our electrical energy storage device & system business. Its research and development division was also subsequently integrated. Thanks to this merger, we are promoting the development of highly competitive products; supported by fusing superior key technologies such as electrical energy storage device development/ manufacturing technology owned by Shin-Kobe Electric Machinery and the basic technology such as material development, evaluation & analysis cultivated ever since our company was founded.

One outstanding feature of our business portfolio is the fact that our company deploys four distinctive electrical energy storage device businesses (lead-acid battery, lithium-ion battery, lithium-ion capacitor and capacitor) as well as owning critical materials to improve the performance of these devices and system technology and products best placed to exploit each device. We set our sights on accelerating development and expanding business; backed by mutual cooperation and a synergy effect (Figure 1). Our company's approach to device and system fields are introduced separately below.

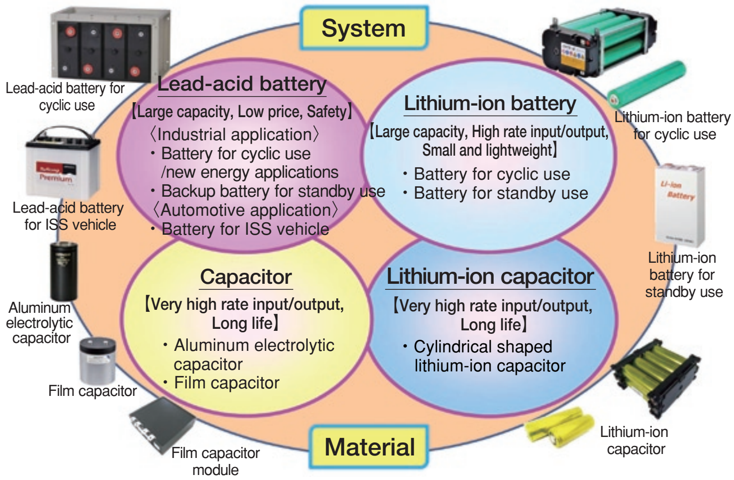



Figure 1 Four electrical energy storage device businesses at Hitachi Chemical

### 2.1 Lead-acid Batteries for Automotive Applications

Our lead-acid battery business is deployed for automotive and industrial applications, and in this section lead-acid batteries for automotive applications are introduced. To reduce carbon dioxide emissions, various measures and technical developments to improve fuel efficiency are being spearheaded, mainly by automobile manufacturers, such as methods to improve fuel combustion, reduce car body weight and downsize in conventional internal-combustion engine automobiles (with gasoline and diesel engines) as well as changing drive systems e.g. in hybrid, pure electric and fuel-cell vehicles. Although hybrid cars have significantly improved their fuel efficiency, they remain costly, so we predict that they will be limited to Japan and North America in the immediate future. As for internal-combustion engine automobiles, which we expect will continue to comprise the majority auto market share, automobiles with ISS (idling stop system) are increasingly attracting attention. ISS can only be implemented by redesigning certain parts such as electric generator, starter and lead-acid battery. However, it boosts fuel efficiency and economy, hence our prediction that sales of ISS-equipped automobiles will expand, mainly in Europe and developing countries, and exceed 35 million automobiles (5 times the current sales volume) by 2020, comprising 30% of total global automobile sales[2]. Automobiles with ISS frequently stop and start engines while awaiting traffic signal changes, supply electric power from the battery to electrical components while the engine is shut off, and enable swift electric power regeneration-type charging using high-powered alternators during traveling. Lead-acid batteries, meanwhile, are prone to be insufficient charging state due to frequent short-term charging and large discharge. Accordingly, there is a need to enhance the regenerative charging efficiency during deceleration to properly exploit the ISS advantage, namely for a high-performance lead-acid battery with charge acceptance performance enabling an efficient time-effective charge and high durability to withstand repeated charging and discharging. In light cars greater fuel efficiency improvements are obtained with ISS, however batteries mounted in such cars require particularly high performance due to the restriction of the size from limited installation space.

In 2006, our company was first to develop[3] a lead-acid battery with a high charge acceptance performance for automobiles equipped with alternator regenerative control as a kind of environmentally friendly vehicle (reducing engine load and improving fuel efficiency by controlling alternator operation). Leveraging this lead-acid battery, a first-generation battery for passenger cars with ISS was developed in 2010, followed by an enhanced second-generation battery for light cars in 2011[4, 5]. The charge acceptance performance of conventional battery for general vehicle and first/second-generation batteries for automobiles with ISS are compared in Figure 2, and their cyclic durability performances are compared in Figure 3. To improve the charge acceptance performance, the rate-limiting charge reaction at negative electrode should be accelerated. In response, we engaged in development focusing on organic additives and carbon for negative electrodes. We also promoted design improvements for each generation, including efforts to optimize active materials for positive electrodes and reduce the resistance of electrode grid. Consequently, the charge acceptance performance of second-generation batteries was approximately doubled compared to the performance of our conventional battery for general vehicles. For second-generation lead-acid batteries, new carbon additive, with electrical conductivity enhanced to ten times the level for general vehicles was employed for negative electrodes. Durability was also boosted

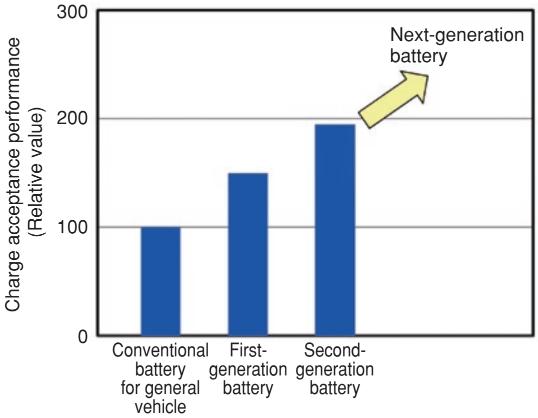


Figure 2 Comparison of charge acceptance performance between conventional and developed batteries (Relative value, based on the value of conventional battery as 100)

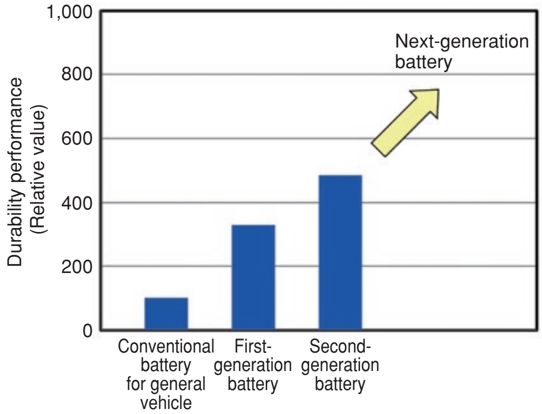


Figure 3 Comparison of durability between conventional and developed batteries (Relative value, based on the value of conventional battery as 100)

around fivefold thanks to successful suppression of stratification (phenomenon of differential concentration between upper and lower layers in the electrolyte solution) and sulfation (accumulation of lead sulfate). These improved high-performance paved the way for us to commercialize lead-acid batteries which can be mounted in light cars allowing battery use under severe conditions of requiring a depth of discharge exceeding 10% compared to passenger cars[5]. We will continue the ongoing development of new technologies, including new separator structures (see details in this technical report) to further improve battery performance.

A lead-acid battery is a device with a chemical reaction integral to the performance development, for which controlling the cell reaction by materials is key. As for development of the above mensioned negative electrode additives, we sought materials which could be controlled to optimize the configuration and physical properties of products involved in the charge/discharge reaction of negative active materials, which significantly impact on battery performance. During this research works, we elucidated[6] the effect of additives and the effect-inducement mechanism by observing morphological change in the active material during the charge/discharge reaction directly *in-situ* and the electrical conductivity of reaction products by atomic force microscopy (AFM), in cooperation with Hitachi Research Laboratory, Hitachi, Ltd. Figure 4 shows examples[3] of observations, revealing clear differences in the amount and configuration of lead sulfate ($PbSO_4$) and lead (Pb), caused by the difference of additives. The elucidation of mechanisms and product development could eventually be accelerated, allowing changes of material on the same place to be observed on the spot, while the effect of additives could also be confirmed more clearly. As for the development of organic additives, we will continue to further improve lead-acid battery performance by developing works backed up by synergistic effects between our groups, such as linkage with material design techniques using quantum chemical calculations and material synthesis/analysis techniques owned by our material group.

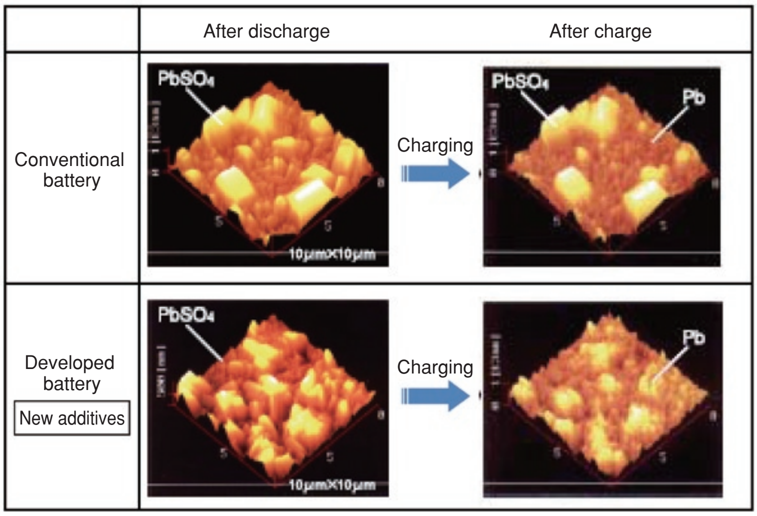


Figure 4 Images of *in-situ* AFM observations which visualize changes in morphology among electrode active materials during charge-discharge cycles

For automotive systems, we foresee progressive applications of advanced driver assistance systems such as collision avoidance for high safety and self-driving as well as efforts to date to improve fuel efficiency. Meanwhile, we must be ready to accommodate increased demand for electric power and secure redundancy where problems affect the electrical supply. We will continue striving in future, as well as to increase storage capacity and improve the reliability of our lead-acid batteries, to search for other possibilities such as innovative lead-acid batteries with significantly improved performance and hybridization with other power sources such as lithium-ion capacitors.

## 2.2 Lead-acid Batteries for Industrial Applications

In the field of lead-acid batteries for industrial applications, we are marketing our products by focusing on applications to back up the power source for important social infrastructure such as hospitals, public facilities, fixed and mobile base stations, data centers and power stations as well as offices and factories to date, leveraging advantages such as low price, safe, large capacity

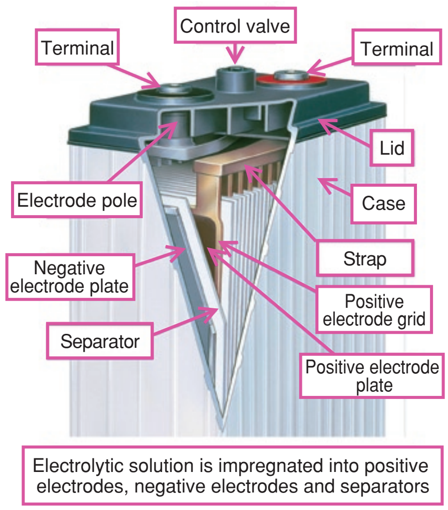
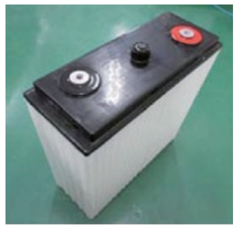



Figure 5 Structure of valve-regulated lead-acid battery

Table 1 Comparison of specifications and appearances between LL1500-WS (new product) and LL1500-W

| | | LL1500-WS (new product) | LL1500-W |
|---|---|---|---|
| Single battery | Photos of exterior appearance |  |  |
| | Number of terminals | Positive electrodes: 3<br>Negative electrodes: 3 | Positive electrodes: 1<br>Negative electrodes: 1 |
| | Nominal voltage-nominal capacity | 2 V−1,500 Ah (10 HR, 25℃) | |
| | Maximum discharge current | 900 A | 600 A |
| | Maximum charge current | 450 A | 300 A |
| Expected life*1 | | 17 years for the application to mitigate fluctuation (25℃)<br>[4,500 cycles at 70% depth of discharge] | |
| Example system* | 1 MW×1 h discharge | Battery capacity 3.8 MWh | Battery capacity 4.6 MWh |
| | 1 MW×0.5 h discharge | Battery capacity 3.1 MWh | Battery capacity 4.6 MWh |

*A representative example, and its performance level may vary depending on specification details and operating conditions.

and easy operation of lead-acid batteries. In addition, we are enhancing and expanding our business in the field of cyclic use applications such as electrical energy storage systems, which store surplus electric power overnight and discharge it during peak daytime hours, electrical load leveling (peak shaving/shifting) and mitigating fluctuations in output power from renewable energy power generators. For these applications, valve-regulated lead-acid batteries are used by exploiting the advantage of long life, high reliability and low maintenance requirement of elimination of water-refilling. In this section, we introduce our approaches to improve the valve-regulated lead acid battery by extending its life and enhancing its product performance in cyclic use.

Conventional valve-regulated lead acid batteries for standby use were difficult to use for cyclic use applications due to the short cycle life of 200 to 500 charge/discharge cycles. Accordingly, we developed the LL series[7] batteries for electrical energy storage applications with an expected life*1 of 3,000 cycles by significantly improving cycle life performance in 2001. In 2005, we developed[9] the LL-S series[8] extending the expected life of 4,500 cycles, followed in 2009 by the LL-W series batteries with an expected life (industry-wide longest life*2) of 17 years for applications to mitigate output power fluctuations from renewable energy generators.

The structure of the valve-regulated lead-acid battery is shown in Figure 5. During the aforementioned development, we achieved the extension of life by undertaking various measures, not only improvement of the main battery components such as the active materials used for positive and negative electrodes, reviewed the alloy composition and shape design of the positive grid, and optimized the specifications of the electrolytes and separator but also optimizing the battery-charging condition, horizontal placement of the electrode plate (see the top-left photo of the lead-acid battery module for cyclic use in Figure 1) and improving materials for the durable battery housing (case and lid) sufficiently to extended life[7)-12)]. Moreover, in September 2014, we could successfully commercialize[13] a high rate charge/discharge cycle performance type battery of the LL1500-WS series, the charge/discharge performance of which became 1.5 times higher than LL1500-W without impairing the long life by reviewing the structure of the battery terminal and strap electrodes, and suppressing heat generation and decline in voltage during the high-current discharge. The characteristics of the LL1500-WS are compared to LL1500-W in Table 1. As the system example shows, a higher performance, that is higher discharge current per battery unit, can be met for systems requiring a high current discharge within a short time with fewer batteries, and realize more economical, compact and lighter battery units. We will also continue work to develop and further enhance battery performance and expand applications for large capacity lead-acid batteries.

During developments to extend life and enhance performance as aforementioned, electrode design technology realizing the suppression of corrosion deformation of positive electrode grids and heat generation at straps, poles and terminals by design optimization was one of the key technologies. Our company has been promoting CAE (Computer Aided Engineering) to design electrodes; targeting design sophistication and reduced designing periods in cooperation with Hitachi Research Laboratory, Hitachi, Ltd. This eventually spawned our unique simulation technology which is capable of predicting the current collection properties (voltage drop, current distribution) on grids, the corrosion deformation of electrode grids, and even manufacturability (molten metal flow, solidification) of designed grids during the casting process[12), 14)]. Figure 6 compares the simulated and actual corrosion deformation results of positive electrode grids, faithfully reproducing curvature deformation and the degree of

*1 Expected life: Life estimated under our recommended operating conditions
*2 Industry-wide longest life: as end of August 2014 and as per our survey

deformation of exterior frame grids. This helped expedite the design optimization of electrode grids and accelerate product development.

The deterioration modes of lead-acid batteries differ depending on service conditions, such as backup applications where batteries await while fully charged (standby), cyclic applications where batteries undergo frequent and repeated charging/discharging, and the same cyclic applications but to store electrical energy and mitigate fluctuations in output power. For example, when mitigating fluctuation of output power from wind power generators, batteries must remain in a state of charge acceptance to respond to fluctuating generated power. Accordingly, this means batteries for cyclic applications are used only in partially (rather than fully) charged state, imposing very severe service condition for the use of lead-acid batteries. When improving performance in terms of extending the battery life, robust deterioration analysis and prediction based on both research & analysis of batteries with long-term actual operation results and simulation tests assuming various operation modes will be important, and it will be crucial to accumulate these data and this expertise. Our group have already been delivered these products to wind power generating plants for use in mitigating fluctuations of output power since 2002[9), 15)] and many data have been accumulated in this field. Our group will continue developing distinctive products by sophisticating service life prediction technique using these accumulated database.

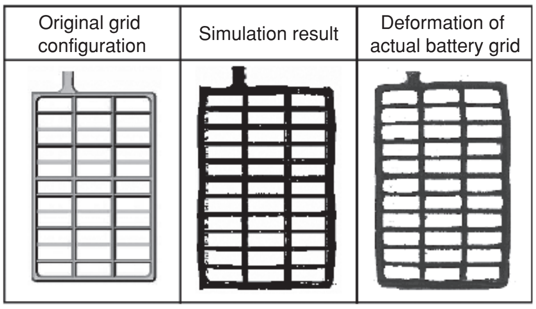



Figure 6 Simulation result of the corrosion deformation of a positive electrode grid compared with the deformation of an actual battery grid

## 2.3 Lithium-Ion Batteries for Industrial Applications

The lithium-ion battery can be featured for small and light weight, large capacity and high rate input/output, and it is widely used primarily for small consumer devices. Similarly, in automotive applications, the lithium-ion battery has achieved full-scale practical and widespread use for various environmentally-friendly vehicles in response to increasing environmental awareness and alongside the demand for high fuel efficiency. Conversely, as for industrial applications, the market for lithium-ion batteries is expected to grow in terms of future applications for power supply backup during emergencies, drive /energy regeneration for railroad vehicles and other industrial machines, load leveling, mitigation of output power fluctuations from renewable energy generator and power grid stabilization.

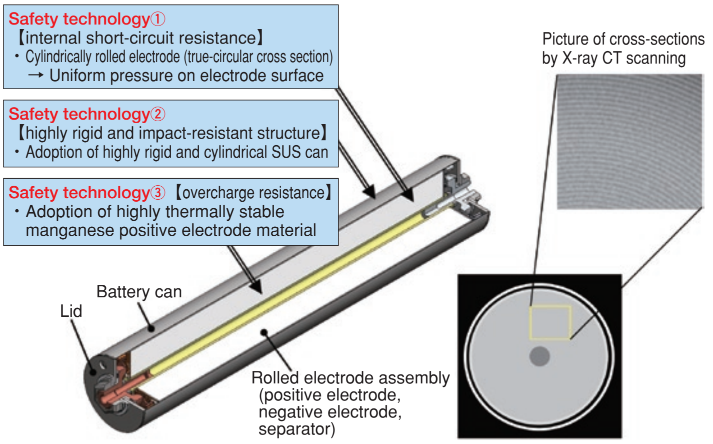
We have been promoting a lithium-ion battery business targeting industrial applications and focusing on two types of battery in the device development process. One of which is for cyclic use applications, featuring repeated charge/discharge cycles and the other is for standby (backup) power supply in emergencies, involving waiting for extended periods in a fully charged state. Our lithium-ion battery can be characterized by its design, which satisfies both large capacity and safety/reliability. Thanks to its large unit cell capacity (single battery); the required cell number can be reduced to form the intended system capacity. This means the total parts count, including peripheral components, can be dramatically reduced and allows overall system reliability to be improved.

As for batteries for cyclic use applications, to date, we have developed lithium-ion batteries used for electric vehicles (EV) and hybrid vehicles (HEV) in cooperation with Hitachi, Ltd. and succeeded in commercial vehicle applications as a world first[16)-18)] in 2000. In 2004, development and manufacturing operations of lithium-ion batteries for automotive applications were transferred to Hitachi Vehicle Energy, Ltd., a joint company established by Hitachi, Ltd., Hitachi Maxell, Ltd. and Shin-Kobe Electric Machinery, Ltd. and we have been engaged in developing large-capacity batteries for industrial applications from 2009. We are developing batteries characterized by their large size & cylindrical shape; a configuration nurtured over many years of battery development for automobiles. On this occasion, we developed a new type of lithium-ion battery CH75, which shows excellent safety, high output power and an extended cycle life, even if its ampere-hour capacity is as large as 75 Ah. The specifications of our developed CH75 cell and its exterior photo are shown in Table 2, with three main technical points to enhance safety (Figure 7). The first was the adoption of cylindrical geometry and the true-circular cross-sectional shape of rolled electrode assembly, which helped ensure uniform pressure distribution over the electrode surface and eliminate internal short-circuit factors caused by hetero-structure, thereby increasing internal short-circuit resistance. Furthermore, this structure can reduce the strain caused by expansion and contraction of the electrode during repetitive charging and discharging cycles and the CH75 realizes the expected 10,000 cycle life[*1]. As a second point, the CH75 was built in a highly rigid cylindrical SUS can, which may prevent expansion during battery operation and provide a highly reliable structure, less prone to damage from external impact. The third point was the adoption of manganese positive electrode material, which shows excellent heat stability and thanks to which a battery capable of meeting safety requirements for industrial-use lithium-ion batteries (JIS C8715-2) could be developed. This CH75 was mounted into the 1 MW container-type electrical energy storage system “CrystEna” developed by Hitachi, Ltd. and verification tests of the ancillary (frequency regulation) service in the power grid stabilization project in the North America are underway[19)].

In recent years, with larger information and communication capacity at higher speed, the power consumption of communication equipment and the electricity required to emergency power supplies for these facilities have increased. Conversely, it has also become increasingly difficult to expand electrical energy storage systems using lead-acid batteries due

Table 2 Specifications and appearance of a lithium-ion battery CH75 cell for cyclic use

| Battery | | CH75 | Photo of exterior appearance |
|---|---|---|---|
| Nominal voltage | | 3.7 V |  |
| Nominal capacity | | 75 Ah | |
| Current | Discharge | Continuous：225 A<br>Maximum：300 A | |
| | Charge | 225 A | |
| Weight | | Approx. 3 kg | |
| Dimension | | Φ 67×410 mm | |
| Expected life*1 | | 10,000 cycles | |



(a) Model cross-sectional drawing in an axial direction (b) Model cross-sectional drawing along the radial direction of a cell

Figure 7 Safety technologies of a lithium-ion battery CH75 cell

to the limited installation space in urban data centers, based on which the compact lithium-ion battery with high volume energy density has emerged as a promising replacement. Data center requirements for emergency backup on a large scale with higher current for about 10 minutes in emergencies have also emerged, with increased amounts of data until the emergency generator starts to operate. The KL200 lithium-ion battery, with capacity of 200 Ah (expected life*1:10 years)[20] was developed for long-term high-capacity backup power systems, while a new TH100 battery, capable of rapidly discharging high electric current, was developed for short-term high-current backup power systems.

Since batteries have always been used in their fully charged state for standby (backup) use applications, we have to assume certain circumstances for lithium-ion batteries leading to thermal runaway caused by internal short circuits or overcharging due to charging device malfunctions, however remote the possibility. Further emergency power supply apparatus is often installed inside buildings in urban areas, for which high-level safety should be required. In this context, working alongside NTT FACILITIES, INC., we have been engaged in flame retardation of electrolytes as a safety technology and to develop lithium-ion batteries for use in standby applications. During this development process, we faced a major technical challenge in balancing flame retardation with extended life. We developed[21] a self-extinguishing electrolyte equivalent to UL94-V0*3 by adding phosphazene flame retardants to flammable organic electrolyte for flame retardation. Lithium-ion batteries have been primarily used for cyclic applications and research into standby applications has been insufficient. It is a known fact[22] that the capacity of a fully charged lithium-ion battery deteriorates over time if left unattended. Accordingly, we conducted a detailed analysis of the capacity deterioration mechanism and successfully balanced an extended life with non-flammability through measures including improving the manganese positive electrode active material and using new electrolyte composition[20]. When developing the TH100, we achieved both high-current discharge performance and an extended life by reducing electrode resistance, increasing the amount of electrical conductive material in the positive electrode and thinning the electrode, thus enabling rapid high-current discharge. The specifications of the cell and a photo of the exterior appearance are shown in Table 3. TH100 is a large safe battery with 100 Ah capacity, capable of continuous discharging at a maximum current of 500 A for 10 minutes or more.

Table 3 Specifications and appearance of a lithium-ion battery TH100 cell for standby use

| Battery | TH100 | Photo of exterior appearance |
|---|---|---|
| Nominal voltage | 3.7 V |  |
| Nominal capacity | 100 Ah | |
| Maximum discharge current | 500 A | |
| Weight | Approx. 7 kg | |
| Dimension | 153×255×72 mm | |
| Expected life*1 | 7 years | |

## 2.4 Lithium-ion Capacitors

The lithium-ion capacitor is an electrical energy storage device with a positive electrode structure of electric double-layer capacitor and a negative electrode structure of lithium-ion battery[23], characterized by (1) higher operating voltage and higher energy density compared to an electric double-layer capacitor and (2) higher output power density compared to a lithium-ion battery and capable of rapid charge/discharge (high power input/output), (3) capable of one million or more charge/discharge cycles (100% in charging depth at room temperature) (long service life), (4) low self-discharge and (5) high-level safety[24]. By exploiting these features, lithium-ion capacitors are steadily coming into use aiming for main power sources, regenerating power and stabilizing

*3 UL94-V0: Material and product safety standards issued by Underwriters Laboratories Inc. to determine flame retardation of resin. "V0 is a class of flame retardant which self-extinguishes less than 10 seconds after removing the flame". As there were no flame retardation standards for batteries, we evaluated this by referring to the standards for resin.

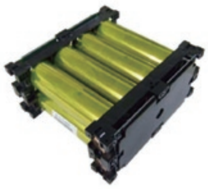
electric power in industrial applications such as automatic guided vehicles (AGVs), stacker cranes[25], construction machines[26] and instantaneous voltage drop compensators[27]. Furthermore, power system stabilization technology using lithium-ion capacitors is being studied to suppress short cycle output power fluctuations from wind power and solar power (PV) generators and deployment to regeneration applications for environmentally-friendly vehicles as a global warming countermeasure in future is expected.

We mass-produced a lithium-ion capacitor featuring a large cylindrical shape in the name of LCAP based on our technologies developed for the lithium-ion battery (see previous section) in October 2009[28]. The lithium-ion must be pre-charged (pre-doped) in the negative electrode during the LCAP manufacturing process. To mass-produce LCAP, we established a technique for pre-charging large-sized cylindrical cells by applying our long-cultivated battery manufacturing technology and successfully achieved commercial production. LCAP shows excellent performance such as high vibration and impact resistance to withstand the harsh vibrations to which construction equipment is subject in operating environments, effective heat resistance at high temperatures and no swelling deformation by overcharging/discharging thanks to its cylindrical structure, true-circular rolled electrode assembly and rigid steel can. We have continued to improve the performance, including capacitance and DC resistance reduction following the aforementioned successful commercialization and our current products are characterized in Table 4.

Table 4 Specifications and appearances of lithium-ion capacitors/LCAP

| Item | SLC-B110A | SLC-B152A | Photos of exterior appearance |
|---|---|---|---|
| Operating voltage range | 2.2～3.8 V | |   |
| Operating temperature range | −15～80℃ | | |
| Capacitance | 1200 F | 2000 F | |
| DC resistance (actual value) | 2.0 mΩ | 1.6 mΩ | |
| Dimension | Φ 40×110 mm | Φ 40×152 mm | |
| Weight | 270 g | 350 g | |

We are developing LCAP monitoring circuit board, packs (modules/packages of multiple cells and monitoring circuit board) and cubicles (electrical energy storage battery boards storing multiple packs and control circuit) by utilizing control and packaging technologies refined through our lithium-ion battery and lead-acid battery development[29, 30]. Representative examples of packs and cubicle are shown in Figure 8. (a) shows a holder-type pack with eight series connected cells, while (b) shows the exterior of a box-type pack containing 40 series connected cells in which 5 sets of 8-cell packs, and a cell controller capable of detecting voltage and temperature, compensating for voltage variation between cells and reporting abnormalities to the host system. (c) shows the inside of a cubicle developed for large capacity electrical energy storage systems, whereby 12 packs of 40 series connected cells described in (b) are mounted inside in four serial and three parallel configurations. This cubicle also mounts a battery management unit, which allows integration of information from cell controllers and reporting the LCAP status to the host system. This cubicle includes a delivery record in applications to mitigate output power fluctuations from solar power (PV) generation.



(a) Holder-type pack

(b) Box-type pack

(c) Cubicle interior photo

Figure 8 Examples of developed LCAP packs and cubicle

## 2.5 Capacitors

The capacitor features a structure with dielectric material inserted between opposed electrodes and is a passive component, which temporarily stores an electrical charge by utilizing the polarization phenomenon while electrical voltage is applied to electrodes. The amount of stored energy involved is less than a battery because it is not accompanied by electrochemical reactions seen in batteries, but the capacitor is a device capable of short-term cyclic charges/discharges and instantaneous large current discharge (high input/output power, long life).

Accordingly, the capacitor has a feature to pass alternating current and not to pass direct current, making it indispensable to share an important role in an electrical circuit to eliminate high frequency noise, transmit signals, and suppress voltage variance and leveling. In recent years, rapid shifting to power electronics/inverter has been underway in various application field in response to reduced power consumption and improved efficiency, and DC capacitors are used as key components for these inverter-control circuit. Such capacitors share the role of eliminating ripple current (pulsating current) and noise superimposed on direct current and further suppressing voltage fluctuation at instantaneous voltage drop by storing electrical charges; which requires high breakdown voltage, large capacity and excellent ripple current dulability.

Our business development efforts have been focused on aluminum electrolytic capacitors and film capacitors. In this section,

we introduce the characteristics and technologies of our capacitors, focusing on high-voltage large-capacity aluminum electrolytic capacitors and film capacitors, which are applicable for the high-voltage inverter-control circuit used in new energy devices such as wind power and solar power (PV) generators, environmentally-friendly cars, railroad vehicles and industrial equipments.

A cross-sectional structure of the aluminum electrolytic capacitor is shown in Figure 9. It has a structure with aluminum metallic film as the positive electrode and an anodic oxide film ($Al_2O_3$) formed on the surface of the positive electrode as its dielectric material, and the thinner the anodic oxide film and the larger the surface area, the higher the electrical energy storage performance. Its dielectric material is a thin film 0.5 to 0.8 μm thick and with breakdown voltage of about 700 V/μm. The surface area has also increased 20 to 40 times due to the micro-textured concave and convex surface patterns achieved by etching; realizing a capacitor with large electrical energy storage capacity per unit volume. Suppressing heat generation by ripple current is a key to improving ripple current dulability. To realize downsizing (a larger capacity per unit volume) and the excellent ripple current dulability of aluminum electrolytic capacitor as required, we have reduced internal resistance and improved[31] the heat dissipation performance of capacitors via surface area expansion thanks to an improved etching technique, modification of the dielectric oxide film, reducing the resistance of electrolytic solution and reviewing the packaging configuration of capacitor elements in a case. Trends of our capacitor performance improvements are shown in Figure 10. Ripple current dulability was more than doubled and approximately 30% downsizing was achieved over the past 20 years.

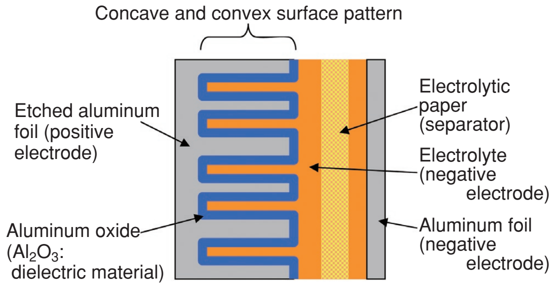



Figure 9 Schematic cross-section of an aluminum electrolytic capacitor

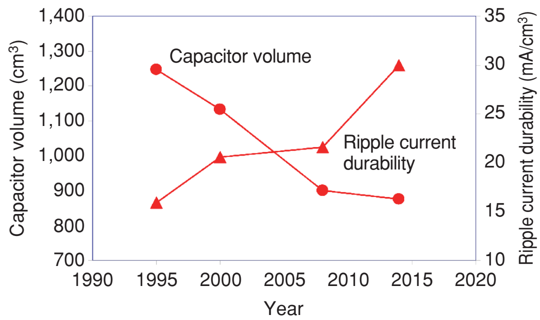



Figure 10 Volume and ripple current durability change of our screw terminal type aluminum electrolytic capacitors (rated voltage of 450 V, 6800 μF)

The film capacitor is using plastic film such as polypropylene (PP) as a dielectric material and features a rolled-up structure of the aforementioned film on which vapor-deposited metals such as aluminum is formed as a electrode (Figure 11). It is characterized by its high breakdown voltage compared to aluminum electrolytic capacitors and small amount of self-heating due to low dielectric loss and small capacitance change by temperature, frequency, etc., which extends its life[32]. We have increased the breakdown voltage, reduced internal resistance and boosted the ripple current dulability by optimizing a vapor-deposited electrode design capable of providing a self-repairing (self-healing) and a self-protection properties, improving device fabrication techniques, such as reducing the stress during film winding process and forming collector electrode (metalicon) and improving the device storing and wiring configuration in the housing[33), 34]. Figure 12 shows photos of the typical exterior appearance of (a) the cylindrical metal case type and (b) module type products developed as high-voltage large-capacity capacitors. The module type of (b) is a capacitor module containing parallel connected assembly of multiple rolled-up film capacitor elements, in a resin case and resin-sealed[34].

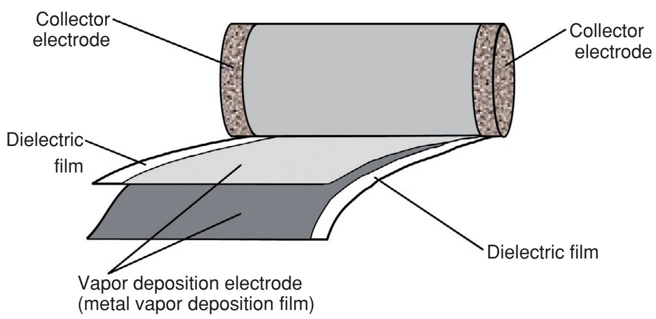



Figure 11 Structure of metallized film capacitor element

(a) 116 Φ Cylindrical metal case-type (b) 300□Module-type

Figure 12 High voltage & large-capacity film capacitors for power electronics applications: (a) cylindrical metal case type, (b) module type capacitor array

## 2.6 Electrical Energy Storage Systems

We have developed various electrical energy storage systems, including emergency power supplies, systems for load leveling (peak shaving/peak shifting) (brand name: Sefla system), regenerated energy storage systems[35] to reuse regenerative electric power from cranes and transportation vehicles and systems to stabilize power grid by utilizing various in-house electrical energy storage devices. The Sefla system is an electrical energy storage system combining lead-acid batteries or lithium-ion batteries with

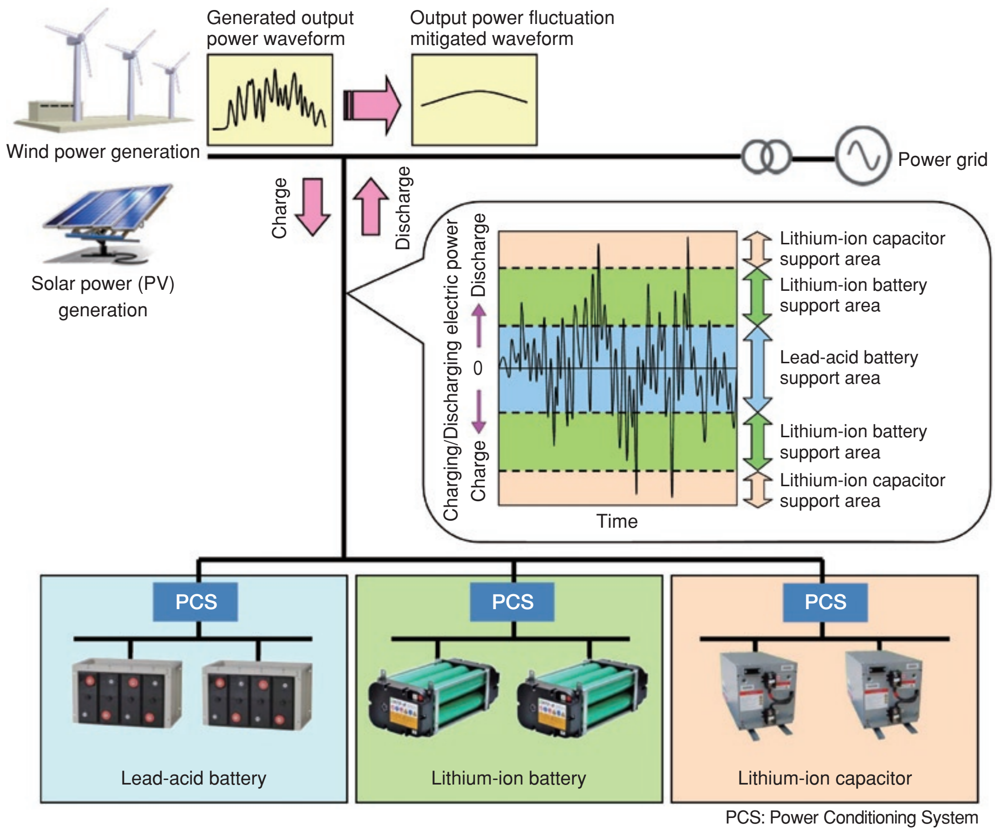



Figure 13 Conceptual diagram of hybrid type electrical energy storage system

a power conditioning system (PCS) capable of shaving the demand power peak in daytime by discharging electrical energy stored overnight and simultaneously facilitating countermeasures to maintain Business Continuity Planning (BCP) as an emergency power supply[36), 37)].

In recent years, the capacity of electrical energy storage systems has become larger-scale; for example, there are 10 MWh and a few MWh level capacities for lead-acid and lithium-ion batteries respectively[38)]. Various requirements are imposed based on the various objectives of use and electrical energy storage system applications, which means responding to such requirements with a single type of energy storage device may not always be appropriate. Each electrical energy storage device has its own unique characteristics and combining multiple types of such devices allows systems to be optimized. We are also considering practical applications of such hybrid electrical energy storage systems, thus ensuring we can offer solutions which balance performance, cost and size by exploiting the performance characteristics of each device. The concept of the hybrid electrical energy storage system is shown in Figure 13. When mitigating output power fluctuations from wind power generators, for example, mitigation of both long- and short-term cycle fluctuations is required. By using both an inexpensive lead-acid battery with excellent relatively slow charging/discharging performance to mitigate long-term cycle fluctuations and a lithium-ion battery or capacitor with excellent fast charging/discharging performance to mitigate short-term cycle fluctuations, downsizing the system and reducing cost is easier to achieve compared to a system configured by a single type of energy storage device. Designing a hybrid electrical energy storage system requires an optimum combination by taking the performance characteristics of each device fully into account. Accordingly, we developed a design tool taking the performance characteristics of various energy storage devices into consideration (refer to this technical report for details of hybrid electrical energy storage systems).

For the operation of electrical energy storage systems, it is important to ensure proper control status of the batteries [chargeable/dischargeable power, state of charge (SOC), temperature, etc.]. As the electrical energy storage system is becoming larger scale and more widespread, the requirement for efficient maintenance has become more urgent. There is a number of issues to be solved; for example, the status of each battery is periodically confirmed by human hands or even if monitoring sensors were mounted on each battery to improve monitoring efficiency, the wired system needed numerous wires, which hindered efforts to ensure the system reliability. We have placed the monitoring system of many batteries into practical applications by measuring the status of each battery with remote wireless slave units and sending data to a master unit[39)] or via the Internet[37)].

## 3 Conclusion

Electrical energy storage device and system products are a group of products which share important roles in comprising infrastructure for a secure and safe society by delivering a steady supply of electricity and achieving a low carbon society. We prioritize environment & energy fields as key business areas and promote our electrical energy storage business.

While we will continue to further improve the performance of each device by developing new technology, economic rationality should also be satisfied for bussiness expansion. Accordingly, we must continue our product development efforts to reduce costs and extend life. We will continue to actively engage in wide-ranging cooperation with internal and external organizations with relevant interests to accelerate our product and application development. We intend to deploy a wide range of product groups and solutions, ranging from materials to devices, right up to systems and services and pledge to follow through with our corporate philosophy, namely “Contribute to society through the development of superior technologies and products that can herald a new era.”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# Lithium-ion Battery System for Smart Grid

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## 1 Abstract

The smart grid has been proposed as an initiative that can be optimized by fusion with IT technology, to control both supply- and demand-side power flow, the response to wide-area power outages due to the decline in power-distribution facilities and power shortages, or system instability after expanding the amount of renewable energy generated and introduced in the USA.

The battery-system roles in the smart grid include load leveling as represented by the peak cut or peak shift, power-system stabilization as an adjustment force and response in emergencies, etc. This report focuses on the contribution of lithium-ion battery system to load leveling alongside renewable energy, stabilization of the supply/ demand, and ensuring lifeline energy in the event of power failure.

## 2 Key Features

・One of the Japan's largest capacity lithium ion battery systems
・Contribution to electric power interchange, demand-supply variation control and securing lifeline in the event of power outages.

## 3 Development Background

Storage batteries for smart-grid operation can be installed together with generators, inside facilities for power system or inside users' facilities as shown in Figure 1. The role of storage batteries in joint installation with generators is to mitigate output fluctuations from renewable energy power generation. The expected role of storage batteries installed inside facilities for power systems is the ability to adjust frequency and provide surplus electricity as a countermeasure. The role of storage batteries installed on the demand side is to prevent demand from exceeding contracted power via peak shaving and peak shifting, and secure a lifeline in the event of power outages[1].

In this report, we report on the Lithium-ion battery system delivered to Kashiwa-No-Ha Smart City as a case example of storage batteries installed on the demand side[2]. In Kashiwa-No-Ha Smart City, AEMS (Area Energy Management System) was introduced to operate, monitor and control energy citywide as a core electrical energy control facility to interchange electrical power between town areas. The expected roles played by the electrical energy-storage system include helping reduce electricity charges, boost the low-carbon society, stabilize the demand-supply energy balance by mitigating output fluctuations from renewable energy photovoltaic generators and securing the minimum electrical power required to sustain the lifeline in the event of a large-scale power outage. Adopting a lithium-ion battery is an effective way of conserving installation space in urban areas by exploiting its compactness and light weight.

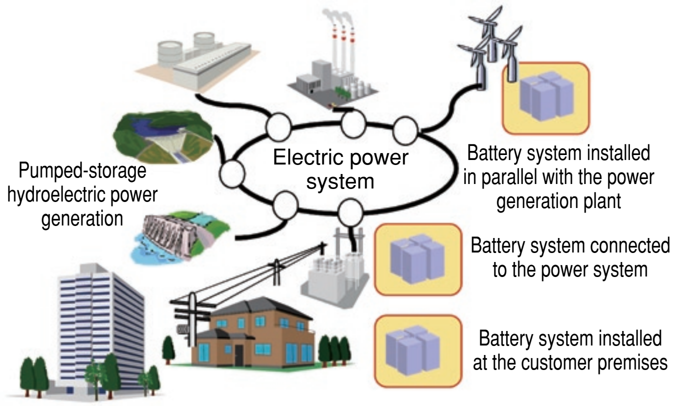


Figure 1 Smart-grid battery system

## 4 Technical Content

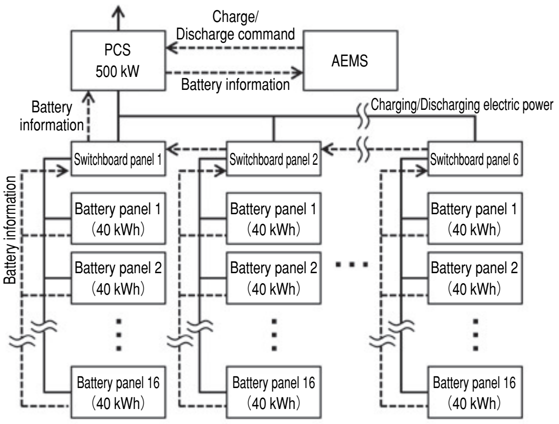


Figure 2 Block diagram of the lithium-ion battery system

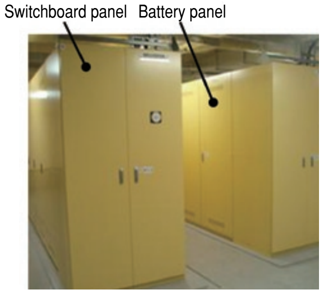


Figure 3 Installed lithium-ion battery system

Figure 4 Lithium-ion “CH75-6” battery pack for stationary use

A block diagram of the lithium-ion battery system delivered to Kashiwa-No-Ha Smart City is shown in Figure 2, while the exterior appearance is shown in Figure 3 and the specification in Table 1. This battery system is capable of storing 3.8 MWh and adopts a layered structure comprising a battery (cell), battery pack, battery panel and switchboard panel. Each switchboard panel acts as an interface with a 500 kW power-conditioning system (PCS), which converts input/output power from a battery system as a DC power source, into AC power. The exterior appearance of the stationary lithium-ion battery pack CH75-6 is shown in Figure 4 and its specification in Table 2.

Table 1 Specification of the lithium-ion battery system

| Item | Specification |
|---|---|
| Input/Output power | 500 kW |
| Battery capacity | 3.8 MWh |

Table 2 Specification of the lithium-ion battery pack

| Item | Specification |
|---|---|
| Type | CH75-6 |
| Nominal voltage | 22.2 V |
| Nominal capacity | 75 Ah |
| External dimension | 228 (width) × 475 (depth) × 151 (height) mm |
| Mass | Approx. 23 kg |

Each battery pack includes a battery assembly housing six cells and a cell controller monitoring the voltage of all cells. Although individual cell voltages may vary after repeated charge and discharge cycles, the cell controller includes a function to detect and adjust the varied voltage automatically to the same level. The cell controller also monitors the temperature of the battery pack.

Twenty-four units of CH75-6 battery packs packed in serial arrangement are mounted in a battery panel. Also, a battery management unit (BMU), which monitors the battery pack status by communicating with the cell controller located in each battery pack, is mounted in the battery panel. The BMU is connected to an master BMU installed inside the switchboard panel and is capable of detecting various problems and malfunctions and adjusting the voltage variance between battery packs as well as transmitting monitored information collected from cell controllers to the master BMU.

Six sets of battery panel blocks - with sixteen battery panels per set - are installed in parallel to comprise the entire system. Meanwhile, the PCS and master BMU communicate with each other and all monitored battery status information is sent to AMES. The master BMU can also operate in degraded operation mode by disconnecting the battery panel in the event of trouble if any one battery panel breaks down, and avoid lowering availability factor.

## 5 Future Business Development

- Developing high output power density lithium-ion batteries in applications to suppress frequency variation by applying next-generation long service life lithium-ion batteries
- Developing high-capacity and energy density lithium-ion battery systems for distribution networks connected to or on-site electrical energy-storage system


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# Flooded Type ISS Battery with Improved High Durability and High Charge Acceptance

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## 1 Abstract

Improved charge acceptance and durability have been required for batteries loaded on the ISS (Idling-Stop System) vehicle, because they have to supply electricity to the vehicle and accept high regenerative power. A new separator design, including a conventional PE separator and specific non-woven fabric, was adopted for the third-generation battery to improve durability to 200% compared to the second-generation ISS battery. We focused on an organic fiber with particular hydrophilic treatment as a non-woven fabric to achieve a new thinner structure and enhance battery performance.

## 2 Key Features

By employing a new separator design which combines a conventional separator with special non-woven fabric, the deterioration of the electrode caused by differences in electrolyte concentrations between the top and bottom of the electrode (stratification)[1] is suppressed and a third-generation battery, which improved the durability of the battery for ISS vehicles by 200% compared to the second-generation product, was successfully developed.

## 3 Development Background

From now, the market for ISS vehicles is expected to grow because although ISS vehicles require little in terms of changing the vehicle system, we can expect[2] it to boost fuel efficiency by 10% or so. Accordingly, we introduced the first-generation battery for ISS vehicles onto the market in 2010, since which time its sales have grown[3]. The ISS battery requires higher durability and charge acceptance than a general purpose battery, to handle the increased electricity discharge during idling stops and regenerated electric power. With this in mind, we improved its charge-acceptance performance by significantly improving the functional capability of the electrode, using positive electrode high-density active material and new carbon for the negative electrode and introduced our second-generation product to the market in 2012[4]. From now, the battery for ISS vehicles will have to be further improved to further enhance fuel efficiency. This time, we worked on a new separator design, which combines a conventional separator with non-woven fabric, together with durability improvement to develop our third-generation product.

## 4 Technical Content

### 1. Regarding stratification suppression with a new type separator design

Figure 1 shows the difference in phenomena between conventional design and a new type of separator design. In the conventional design structure, sulfate ions descend and differentiate the concentration between the top and bottom of the electrode, triggering a phenomenon called stratification. While charging the battery, sulfate ions generated at the electrode have higher specific gravity, which tends to cause them to move downward. At the same time, sulfate ions generated at and then repelled by the negative electrode again move downward, leading to a concentration-stratified layer. In the redesigned separator structure, the downward movement of sulfate ions can be hindered by bonded non-woven fabric on the surface of the negative electrode to prevent stratification.

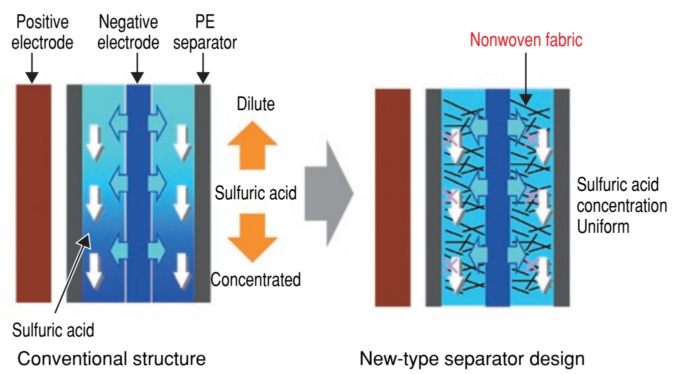


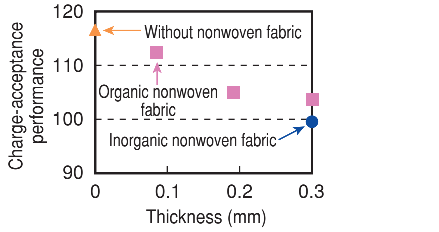

Figure 1 Stratification phenomena of conventional structure and new type separator design

## 2. Assessment of charge-acceptance performance

Table 1 shows a few types of non-woven fabric which we assessed. However, it is difficult to obtain any thinner inorganic non-woven fabric given the strength requirement and manufacturing process, which increases the higher internal resistance of a battery. Conversely, the thickness of organic non-woven fabrics can be altered in many ways, meaning both scope to reduce internal resistance and improve charge-acceptance performance. On this occasion, we applied hydrophilic surface treatment to organic non-woven fabrics to improve their water wettability by coating the fabric surface with fine silicon dioxide particles. Figure 2 shows the effects of various non-woven fabric types on charge-acceptance performance in the new separator design structure, which was improved by thinning organic non-woven fabrics. It is thought that this may have been attributable to easier dispersion of sulfate ions, helped by lower internal resistance due to thinner non-woven fabrics.

Table 1 Type of nonwoven fabric

| | Specification (1) | Specification (2) | Specification (3) | Specification (4) |
|---|---|---|---|---|
| Material | Inorganic nonwoven fabric | Organic nonwoven fabric | | |
| Thickness (mm) | 0.3 | 0.1 | 0.2 | 0.3 |

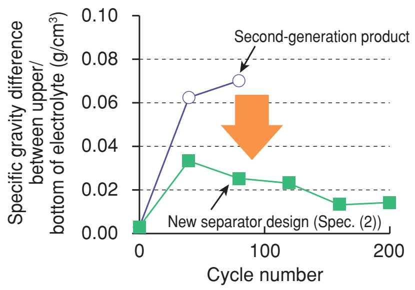



[charge-acceptance performance of inorganic nonwoven fabric as 100]

Figure 2 Influence of various nonwoven fabrics on charge-acceptance characteristics, using a new type-separator design

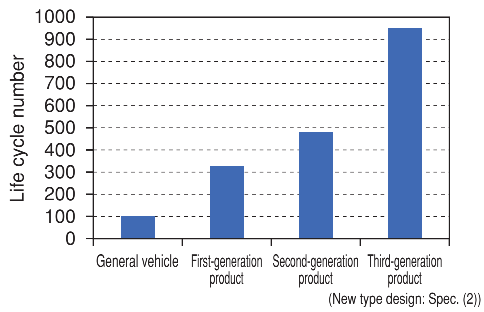
## 3. Assessment of durability

Durability was assessed by measuring the total number of charge-discharge cycles at a charge/discharge depth of the original battery capacity of about 10% to simulate service conditions in ISS vehicles. Figure 3 indicates the stratification level during charging/discharging cycles in the new separator design structure, which was assessed by determining differences in the specific gravity of electrolytes between upper and lower parts. Figure 4 indicates the results of a durability test in the new separator design structure. The durability of the third-generation product was improved by 200% by limiting stratification in the new separator design structure, which is comparable to approximately ten times the durability of general vehicles.




[Life cycle number of second generation product as 100]

Figure 3 Comparison of stratification degree between the first generation and new type separator during cycling




[Life cycle number of general vehicle as 100]

Figure 4 Durability improvement by new type separator design

## 5 Future Business Development

Globalization of the flooded-type ISS battery with improved high durability and high charge acceptance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**【Reference Patents】**

Patent Number 5621841, Patent Number 5598532, Patent Number 5126454, Patent Number 5500315

Hitachi Chemical
Working On Wonders

# Large Format Hybrid Energy Storage System for Power Leveling

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## 1 Abstract

To prevent global warming, renewable energy sources, such as PV (photovoltaic) and wind power, are becoming increasingly popular. Since power generated via renewable energy sources fluctuates depending on the weather, when introduced on a large scale, power leveling using the BESS (Battery Energy-Storage System) is required. However, power-leveling applications also incorporate peak-cut, peak-shift, and power fluctuation suppression, for which different battery characteristics, e.g. in terms of power or capacity, are required. Accordingly, Hitachi Chemical Ltd. has joined Hitachi Ltd. to develop HBESS (Hybrid Battery Energy-Storage System), to provide an optimized combination of various battery technologies and a more compact and economical BESS with suitable capacity and power for the target application.

## 2 Key Features of HBESS (Hybrid Battery Energy-Storage System)

・Optimally combining multiple batteries with different performance characteristics can provide the BESS (Battery Energy-Storage System) with sufficient capacity and power for its targeted applications.
・The capacity, downsizing and service life of BESS (Battery Energy-Storage System) can all be successfully optimized.

## 3 Development Background

In recent years, to counter global warming, renewable energy sources such as PV and wind power generation have been drawing increasing attention as clean energies; eliminating carbon dioxide emissions. Renewable energy is also being deployed on a massive scale through FIT (Feed in Tariff) and concerns about power supply shortages by halting operations of almost nuclear power plants after the Great East Japan Earthquake. However, since power generated via renewable energy sources inherently fluctuates depending on the weather, power systems cannot absorb these fluctuations unaided and it becomes increasingly difficult to provide a stable power supply on a large scale. In actual fact, each electrical power company has started imposing temporary measures to limit the introduction of renewable energy as of October 1, and there is a urgent need to take countermeasures for power fluctuations.

Accordingly, we at Hitachi Chemical are planning the BESS (Battery Energy-Storage System), which includes a peak-shifting function, whereby BESS charges electricity generated by PV in daytime and discharges it overnight as well as a demand-response function by supplying electricity from storage batteries to a power system to control maximum demand in response to temporary peak power consumption occurring every few minutes. However, lead-acid batteries are best suited for peak shifting in terms of capacity and lithium-ion batteries preferable for demand response in terms of output power but no batteries suit both applications, which meant we had to adopt a system-based approach to solve this problem.

## 4 Technical Content

Figure 1 (a) shows the concept of HBESS, the (Hybrid Battery Energy-Storage System). The graph shows sloping lines by plotting power as the relation of power vs. capacity to show performance characteristics, while the red line represents the performance characteristics of power type LIB (Lithium-Ion Battery) and the blue line represents capacity-type LAB (Lead-Acid Battery). To build a BESS which meets the required capacity and power characteristics (☆) of targeted applications, excess battery capacity may be required to replace insufficient LIB capacity and insufficient power from LAB. By combining a capacity-type LAB and a power-type LIB responsible for storage capacity and a LIB responsible for power, the required battery energy can be reduced, allowing a more compact and economical BESS.

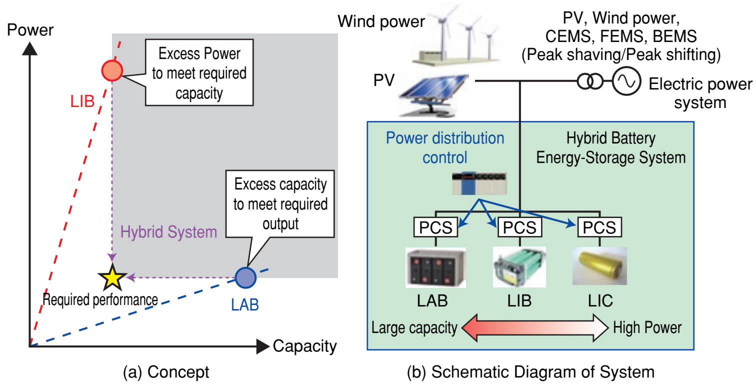


Figure 1 The concept of HBESS(Hybrid Battery Energy-Storage System)

Figure 1 (b) shows a schematic diagram of the HBESS (Hybrid Battery Energy-Storage System). PCS regulates the charging/discharging to batteries and converts electrical energy between AC and DC, while each battery is connected to the grid via PCS. The following steps will also be required to implement the power distribution control: 1) Detect the variation generated in electric power by wind power or PV and calculate the total required charge/discharge power for batteries, 2) Determine the charge/discharge power amount to distribute the calculated amount of power for each battery by taking into account their individual performance characteristics, and 3) Power charging/discharging instruction to each PCS.

Figure 2 shows the preliminarily calculated effect on power leveling for the case of wind power. Figure 2 (a) shows an example of charging/discharging electrical power wave forms in this case, while Figure 2 (b) shows the preliminarily calculated hybrid effects also in this case. Here, a LAB helps level out electric power at low frequencies requiring higher storage capacity, while LIB helps level out power at high frequencies requiring higher power, helping eliminate excess battery capacity. We determined that the potential cost saving allowed for storage batteries through HBESS could reach about 40%.

This preliminary calculation result may also be contingent on wind conditions and the operational philosophy behind the running of systems such as power distribution control between LAB and LIB.

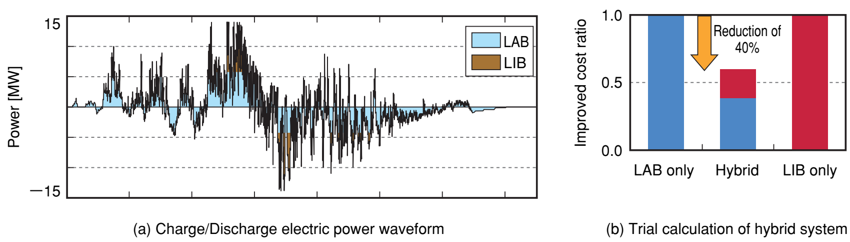


Figure 2 The application example of HBESS for the wind power

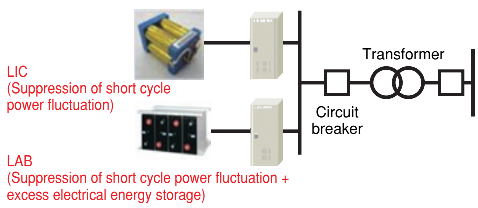


Figure 3 The application example of HBESS by LIC and LAB

## 5 Future Business Development

・Application of our HBESS (Hybrid Battery Energy-Storage System) to NEDO's "Technology Development for Safe, Low Cost and Large-Format Energy-Storage System (2023)" (joint project with Hitachi Ltd.)

【Reference】

1) Takeda, et al.: Design of Hybrid Energy Storage System using Dual Batteries for Renewable Applications, IEEE PES GM (2014)

# Advanced analysis of LIB and Related Materials

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## 1 Abstract

To develop advanced Li-ion batteries, elucidating the influence of functional components on battery performance is crucial. However, Li-ion batteries are very difficult to analyze, because they comprise various organic-inorganic or liquid-solid materials. Accordingly, in this study, we developed a new analytical method to elucidate the two- and three dimensional nanostructure and crystalline distribution as well as a method to visualize the quantified dispersion state of the ingredient for Li-ion batteries.

## 2 Key Features of Analytical Techniques

・New advanced analytical technique applicable to an analytical area.
・Clarification of unidentified performance characteristics and the beneficial effect of lithium-ion batteries and battery related materials.

As specific successful case examples:

1) 3D image-surface observation: Visualization of coating formation, acicular structure and fibrous (mesh) structure in nanoscale.
2) Visualization of images starting from the spatial distribution of functional groups to the distribution of binders inside the electrode.
3) In-situ observation of the microstructure inside a battery used for analytical study during charge/discharge cycles using a high-resolution X-ray CT apparatus.

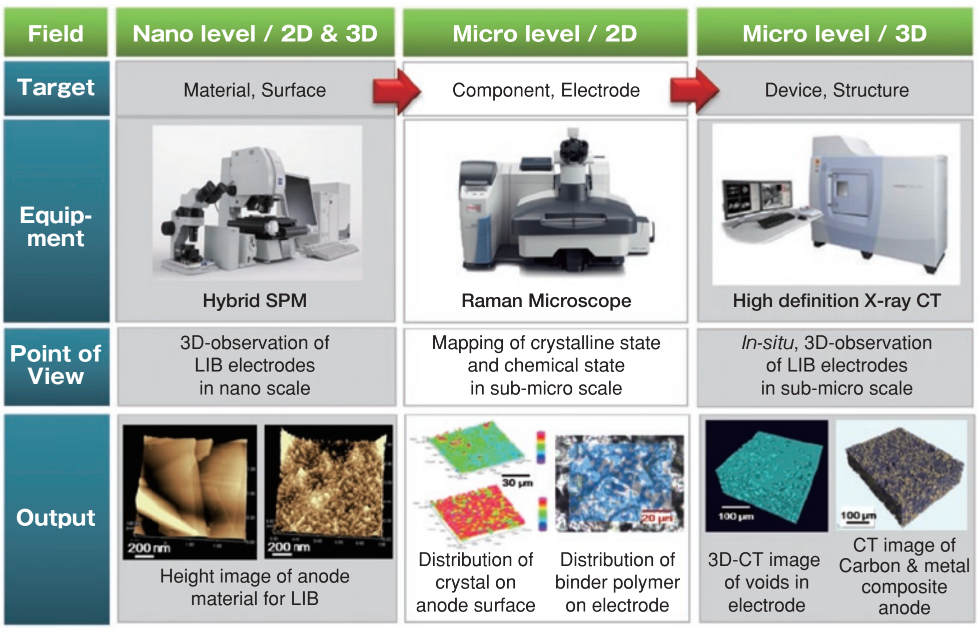


Figure 1 New analytical technologies suitable for various fields

## 3 Development Background

LIB needs a breakthrough to boost energy density, service life and safety while reducing costs, hence the need to develop new technology, including new material. As well as overcoming current technological challenges, various development works are ongoing to realize characteristics tailored for specific applications. However, it is difficult to analyze LIB performance by traditional analytical methods alone because the analytical target is an organic/inorganic and solid/liquid composite body, the main components of which are complex active material particles with a textured surface, and which also include many unidentified areas. Accordingly, we tried a new analytical approach to clarify advanced performance characteristics in this field.

## 4 Technical Content

During this research, we newly developed complex analytical methods allowing the 2D- and 3D-nanostructure of inorganic/organic and solid/liquid composite bodies, crystalline distribution and clarification of the dispersed state of specific components (visualization, quantification) to be clarified as follows:

① 3D image-surface observation: Visualization of the coating formation, acicular structure and fibrous (mesh) structure in nanoscale.

② Visualization of images starting from the spatial distribution of functional groups to the distribution of binders inside electrodes.

③ In-situ observation of microstructures inside batteries used for analytical study during charge/discharge cycles and using a high-resolution X-ray CT apparatus. Leveraging these new analytical approaches, the manifestation mechanism of the characteristic features of LIB and battery materials was clarified as follows:

1) By visualizing nanoscale morphological differences, we determined essential key factors to improve battery characteristics. Composition of active material, difference in 3D surface morphology, namely, difference in 3D image of smooth structure, acicular structure and fibrous (mesh) structure, were elucidated using scanning probe microscope. This approach could make capturing of 3D morphological image over large area easier while conventional cross-section image by Transmission Electron Microscope (TEM) was difficult to create the same; thereby, capturing nanoscale surface morphology of active material, which is high impact factor for battery performance, was made easier.

2) The difference in the dispersed state of binders caused by differences in the composition of active materials was confirmed by microscopic images, and it was also clarified that the area occupancy rate of the binder was one of factors behind lower resistivity. Figure 2 shows the correlation between the dispersed state inside the electrode and the AC resistance for each binder type. For each binder type, we confirmed that the lower the area occupancy rate, the lower the AC resistance of the electrode. The dispersion state of the binder as an electrical insulating material should occupy the minimum area in the electrode to reduce resistance. By utilizing dispersion state assessment techniques inside the electrode, optimization between the composition of binder/additive and the dispersed state can be successfully implemented and thus help boost electrode performance.

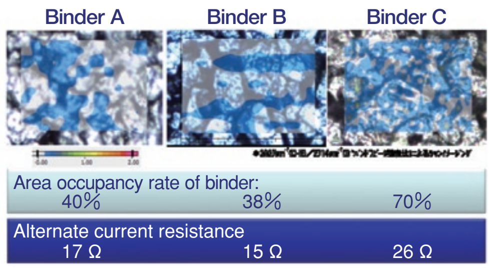


Figure 2 Relationship between distribution in electrode and AC resistance based on the difference in binder type

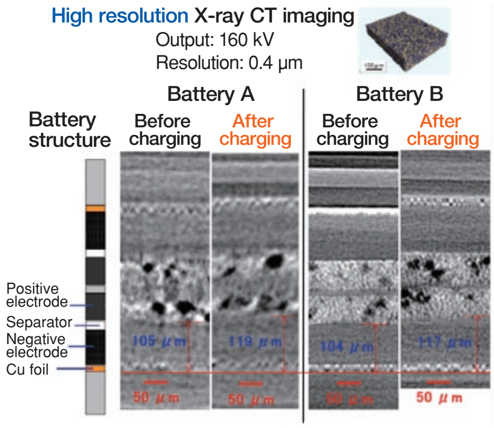


Figure 3 Expansion / constrictive analysis of the battery electrode by X-ray CT

3) Swelling inside the battery during the charging cycle was confirmed by in-situ observation of its internal microstructure during the charging/discharging cycle. Figure 3 shows analytical images of the electrode swelling/contraction inside the battery, as identified by non-destructive X-ray CT scanning. The negative electrode layer swelled by 12 to 14 μm after charging and approximately in line with the theoretical expansion coefficient of graphite. From now, higher durability and lower swelling of electrodes should be essential to extend LIB service life. We expect that the non-destructive visualization measurement technique will play a greater role in microscale observation inside LIB during the charging/discharging cycle.

## 5 Future Business Development

・Technical developments in the field of various battery energy-storage device types and their related materials by applying this analytical approach

・Detailed elucidation of the characteristics and manifestation mechanism of inorganic/organic and solid/liquid composite materials in infocommunication and life science fields


【References】

1) Tetsuo Sakai: Material Integration, Vol. 23, No. 06, pp. 1-11, TIC (2010)

2) 2010, Fact-Finding General Survey of Battery & its Related Market (second volume), Fuji Keizai (2009)

Hitachi Chemical
Working On Wonders

# Reusable Thermal Conductive Sheet Containing Vertically Oriented Graphite Fillers"TC-S01A"


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## 1 Abstract


As the performance of semiconductor package improves and they become increasingly compact, the need to control heat through thermal interface materials has become more and more important. To meet the demands for thermally conductive and flexible material, we have developed and commercialized a high-performance solution. By orienting graphite fillers vertically within an acrylic rubber-based matrix, the TIM(Thermal Interface Material) we developed provides both high thermal conductivity and flexibility.

Generally, a TIM can be applied and used only once. However, we discovered that CPU testing and machine maintenance applications demand ease of reuse. In this report, we will discuss how we developed a metal foil laminated TIM which facilitates removal and reuse.


## 2 Key Features

・Aluminum foil is only applied to one side of the sheet, allowing for one sided reusability.
・Bacause this thermal conductive sheet uses vertically oriented graphite fillers, high thermal conductivity is achieved in the "Z" direction.

## 3 Development Background

We developed the TIM (Thermal Interface Material) TC-001, containing vertically oriented graphite fillers in an acrylic rubber-based matrix, by applying our composite and structural control technology to combine these two materials effectively. Figure 1 shows an image of a cross-section of TC-001, revealing the vertically oriented large-particle graphite creating a pathway to the other side in TC-001. This structure has achieved 90 W/m・K thermal conductivity in the thickness direction.

To apply TIM (Thermal Interface Material), it should be sandwiched between the heating element and heat sink to boost heat transfer efficiency. However, it should also be repeatedly used to improve productivity in Burn-In Tests as an initial characteristic confirmatory test for CPU'S. When it is necessary to exchange CPUs or power modules used in electronic devices, general purpose grease may cause Burn-In and exchanging such parts for repair can be extremely difficult.

In this context, we attempted development work to impart reusability while maintaining the high thermal conductivity of vertically oriented graphite based TIM.

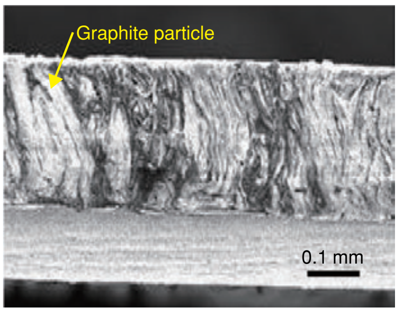

Figure 1 Cross-sectional image of TC-001

## 4 Technical Content

### (1) Design concept to impart reusability

Figures 2 and 3 show the cross-sectional structure and exterior appearance of TC-S01A, respectively. TC-S01A uses soft aluminum foil, which can prevent oxidation degradation by forming a passive state. In combination with TIM with vertically oriented graphite, TC-S01A realizes, ① high thermal conductivity (15 W/m·K, including contact resistance), ② No sticking to the heating element, and ③ Flexibility.

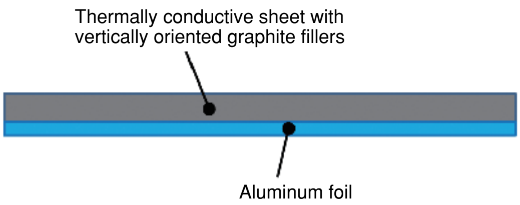


Figure 2 Structure of TC-S01A

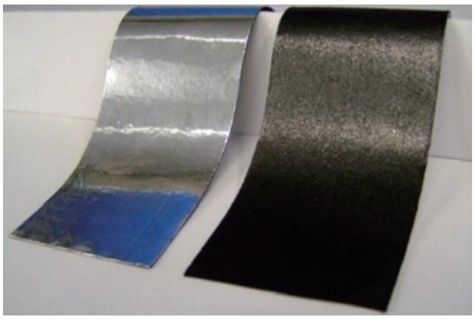

Figure 3 Appearance of TC-S01A (Left) Aluminum foil side (Right) Sheet side

### (2) Assessment of reusability

Figures 4 and 5 show the stress-strain curve and thermal resistance change after 1000 cycles of the TC-S01A compression recovery test. TC-S01A can show a reduction in curbed thickness and additionally retained recovery properties after repeated stress in the direction of vertically oriented graphite. It was also confirmed that thermal resistance levels did not worsen. TC-S01A excels in terms of reusability under harsh temperature conditions and is expected to become a main component in semiconductor applications including Burn-In Inspections and electronic devices such as power modules.

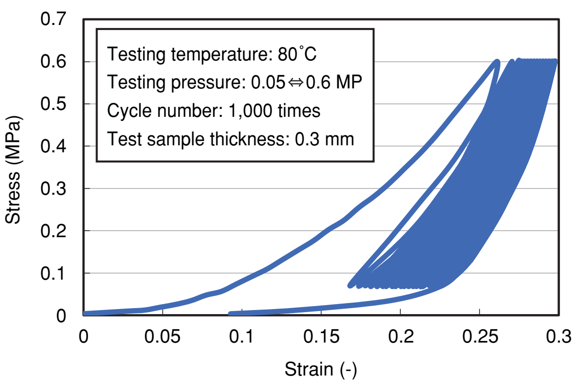


Figure 4 Stress-strain curve

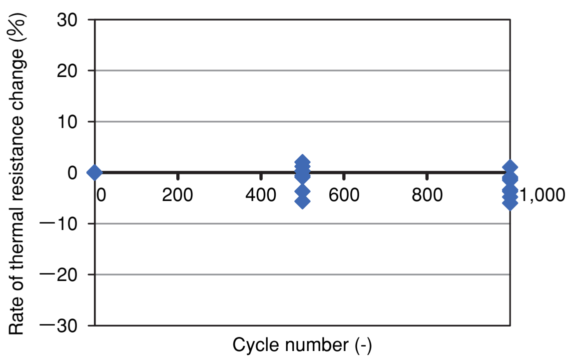


Figure 5 Change of thermal resistance with compression test

## 5 Future Business Development

・Development of TC-S01A for various applications at home and abroad


【Reference】

1) Hitachi Chemical Technical Report No. 53 (2009-10)

# Wavelength Conversion Particle

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## 1 Abstract

Among the many kinds of renewable energy, photovoltaic power generation has shown remarkable growth and is rapidly penetrating. The crystalline Si photovoltaic method is mainstream and its production volume is expected to increase steadily in future. However, the unit price for generated electricity must be reduced to facilitate the further penetration of photovoltaic power generation, and improving conversion efficiency and cutting costs have become urgent priorities for PV manufacturers and material suppliers. We developed Wavelength-Conversion Particles (WCP); applicable to PV module encapsulation sheets of the PV module and capable of reducing loss due to the spectrum mismatch between the sensitivity of the crystalline Si cell and sunlight, and achieving higher conversion efficiency.

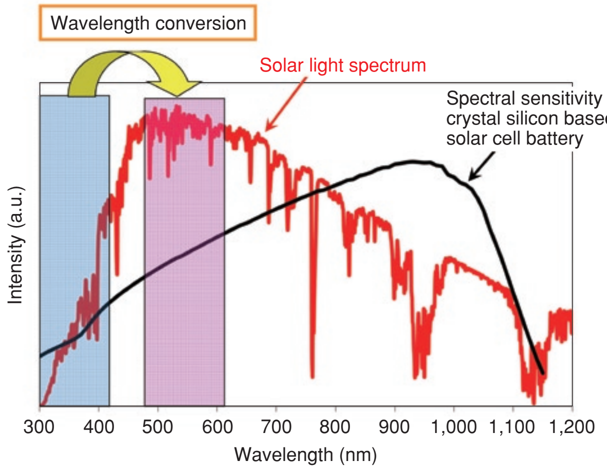


Figure 1 The solar spectrum and spectral sensitivity of the silicon crystal

## 2 Key New Product Features

- High wavelength conversion efficiency, providing long-term durability for PV modules.
- Good dispersibility in EVA sheets as well as retaining initial transparency, achieved by the composite particles consisting of acrylic resins and specialized phosphors.
- Existing PV module assembly lines can be adapted.

## 3 Development Background

In 2007, to utilize sun light effectively, we focused on fluorescent material capable of converting short-wavelength light (ultraviolet rays) to long-wavelength light (visible light) and started research work. In 2011, we analyzed the durability and optical scattering effect etc. and found WCP, which was phosphor contained inside acrylic resin particles, effectively improved wavelength-conversion efficiency (Figure 2).

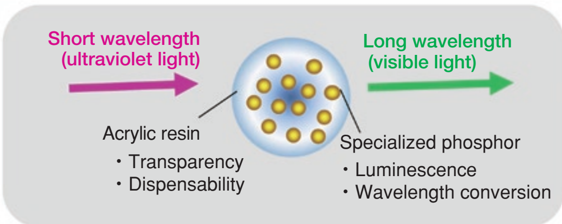


Figure 2 Acrylic resin capsules enhance the wavelength conversion

## 4 Technical Content

Table 1 and Figure 3 show the performance characteristics and exterior appearance of WCP respectively.

To start WCP development, we turned our attention to the excitation spectrum, phosphor intensity and fluorescence quantum efficiency of phosphor, sunlight spectrum, and spectral sensitivity of crystal silicon-based solar PV cells. Consequently, we found the phosphor effective in converting UV photons efficiently and improving the efficiency of crystal silicon-based solar PV cells. We also optimally exploited our own fine particle manufacturing technology to encapsulate phosphor with acrylic resin and adjust the diameters, distributions and in-capsule concentrations of its particles. WCP can be applicable to most currently used encapsulation sheets, including EVA (Ethylene-Vinyl Acetate), which is the mainstream option as well as olefin, ionomer and polyvinyl butyral-based resin sheets. Moreover, a wavelength-conversion function can also be imparted to the encapsulated sheet without a sheet manufacturing process by compounding WCP with other additives during the sheet manufacturing process. The solar PV battery module using an EVA encapsulation sheet as a light-receiving surface-side encapsulation material, which was imparted with the wavelength conversion function, improved conversion efficiency by a relative value of 2.2% compared to that using a conventional EVA encapsulation sheet. A reliability assessment result also showed a level equivalent to that using conventional EVA encapsulation sheets. Sales of this WCP material as WCP-I commenced in the first half of 2014 and were commercialized by one encapsulation sheet manufacturer (Figures 4 and 5).

Table 1 Property of Development Product

| Item | Unit | WCP |
|---|---|---|
| Excitation wavelength | nm | 300–400 |
| Emission wavelength | nm | 500–600 |
| Emission color | — | Green color |
| Particle diameter | μm | 90–110 |

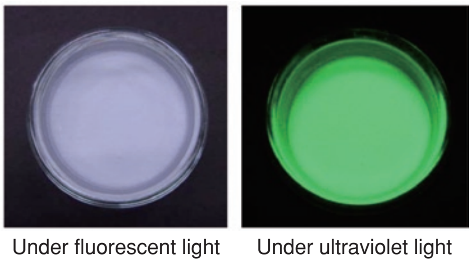


Figure 3 Appearance of wavelength conversion particle

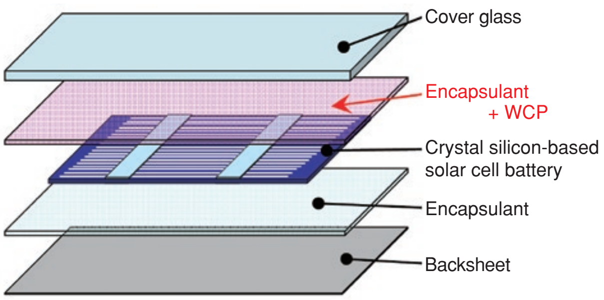


Figure 4 The photovoltaic module using WCP

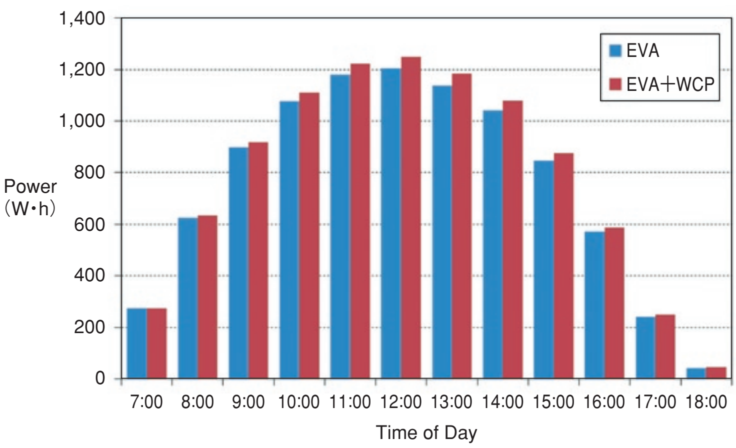


Figure 5 Exposure test of PV module with WCP

## 5 Future Business Development

- Development of next-generation high efficiency wavelength-conversion material
- Development of new applications to utilize the wavelength-conversion function (anticounterfeit, authenticity discrimination, optical element, etc.)

**【Reference】**

1) 2014, Solar Cell Battery and Related Technology, Prospects of the Current and Future Market, Fuji Keizai (2014)

# Concept & Situation of Open Laboratory

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## 1 Abstract

With the performance of electronic devices soaring in recent years, the miniaturization, high density and structural complexity of semiconductor packages (hereinafter, the package) are all progressing, while the product cycle has been shortened. Accordingly, it has become even more important to develop novel materials and provide them to customers on time. To propose integrated solutions to our customers, we established the Open Laboratory in our Packaging Solution Center to implement total assembly-process solutions, including the development of advanced packaging technologies and proposal of materials.

## 2 Concept of the Open Laboratory

Ahead of the rest of the world, we set up the Jisso Center to evaluate and analyze semiconductor packaging material internally and have promoted the development of various packaging materials since 1994 (Figure 1). Consequently, we expanded our product lineup, including semiconductor materials, from pre- to postprocessing.

In parallel with ongoing exponential functional improvements in electronic devices, as reflected by smartphones and tablet PCs in recent years, the miniaturization and densification of semiconductor packages has rapidly progressed. In response, the semiconductor packaging structure has become more complex, including not only high-density surface packaging but also a 3D structure with TSV(TSV: Through Silicon Via) and the packaging process had also been varied (Figure 2). While the product cycle has been shortened, it has become even more important; not only to propose novel packaging materials and provide them to customers on time but also promptly propose integrated solutions to customers, including advanced packaging technologies and from their perspective.

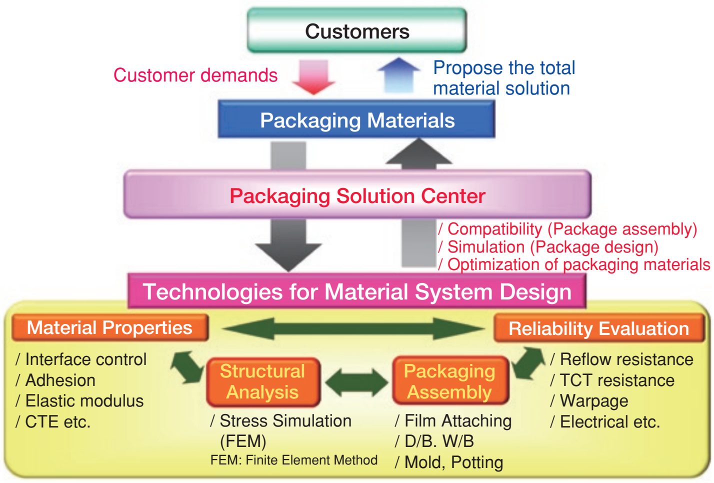


Figure 1 Activities of Packaging Solution Center

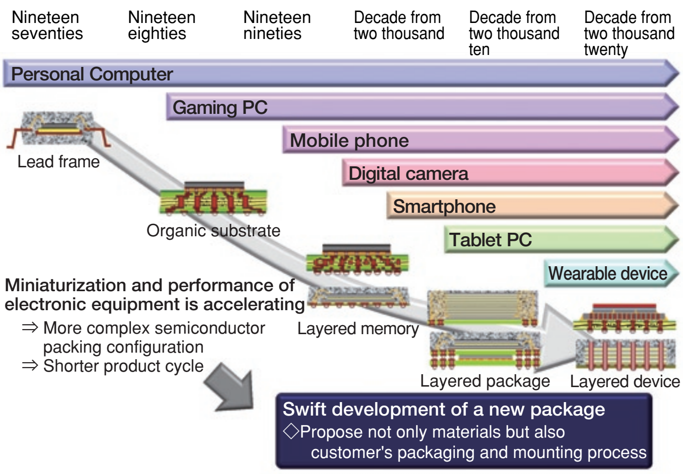


Figure 2 Trends in electronic equipment & semiconductor package

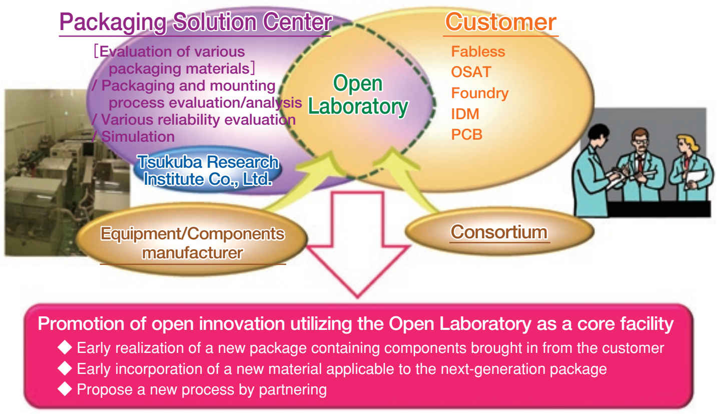


Figure 3 Open Laboratory Concept

Subsequently we established the Open Laboratory in our Packaging Solution Center (former Jisso Center), which saw us introduce state-of-the-art packaging and testing equipment therein based on various semiconductor packaging materials, evaluation and analytical technologies to respond timely to various customer needs (Figure 3). In the Open Laboratory, most advanced semiconductor packages from customers can be packaged and evaluated utilizing our own various packaging material product lines. Also utilizing our Open Laboratory as a core facility, we will promote the development of novel materials and processes by committing to active cooperation with equipment manufacturers, process developers and component manufacturers. Furthermore, we will continue to propose optimized material combinations and processes utilizing our accumulated extensive material database and performing various simulations in response to the next-generation package structure as a design basis.

## 3 Key Features and Activity Situation of the Open Laboratory

The key features of the Open Laboratory can be summarized as follows (Figure 4):

1) Various packaging approaches can be attempted, from ultra-thin chip stack at 40 μm thick or less and fine-pitch flip-chip package 50 μm thick or less; utilizing various state-of-the-art component packaging equipment designed for $\phi$300 mm wafers.
2) Different simulations can be carried out utilizing an extensive material database.
3) Micro-defects can be analyzed using high-precision analytical equipment.

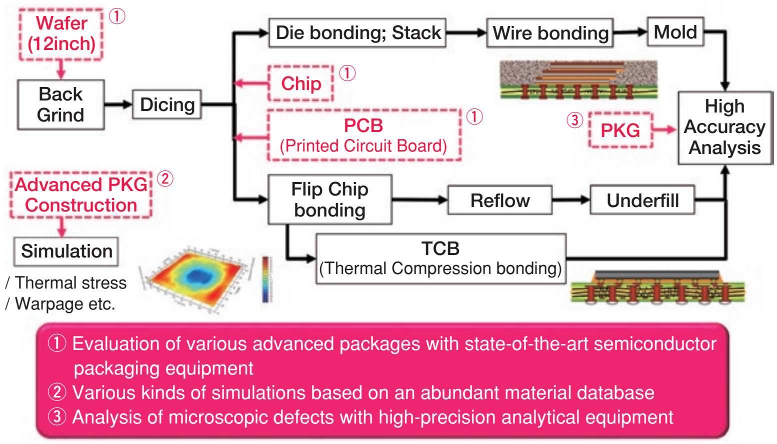

Figure 4 Open Laboratory Activities

Moreover, packaging evaluation can also be performed using various materials for components, such as semiconductor devices supplied from customers, and integrated technical supports from the usual trial and error approach for materials proposed by customers to establish process conditions for customers' packages. This allows us to expedite the required time for evaluation by customers and boost the early materialization of semiconductor packages by customers. Conversely, as for 3D packages with TSV, we joined the IMEC 3D Program and are promoting the evaluation of various packaging materials.

## 4 Future Business Development

1) Early development of a packaging process to handle next generation packages and accelerating material development.
2) Proposal of novel materials and processes to create new value by promoting open innovations with equipment manufacturers, process developers and component manufacturers; utilizing our Open Laboratory as a core facility.

# Low Dielectric Constant Multilayer Material for Mobile "MCL-E-78G"

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## 1 Abstract

Amid the high functionality and high-speed communication of mobile devices such as smartphones and tablet PCs, it has become difficult to control impedance and RF characteristics by increasing wiring density and layer count. Accordingly, related motherboard designs have reached the limits for standard FR-4 material [1), 2)].

We have developed a new low dielectric constant multilayer MCL-E-78G material for mobile devices. As this material shows good dielectric constant, it facilitates impedance control of PWB, meaning this material may provide an improved margin for PWB design compared to standard FR-4 material.

## 2 Key Features of MCL-E-78G

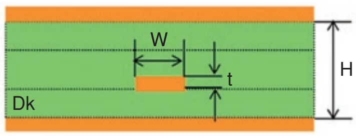
- MCL-E-78G has superior dielectric properties.
- MCL-E-78G has high thermal stability and glass transition temperature (Tg).
- MCL-E-78G is an environmentally-friendly material using halogen-free flame retardants.

## 3 Development Background

Recent years have seen functional improvement in mobile devices, as exemplified by smartphones. Accordingly, it is expected to become more difficult to secure a sufficient margin for PWB (printed wiring board) design due to narrowed wiring space and the increased number of layers. Lowering the dielectric constant of substrates may be one approach to solve this PWB designing problem. By lowering the dielectric constant of substrate, as indicated in Figure 1, a sufficient wiring pattern width can be secured, and an improved margin for designing PWB can be expected.

In the above context, we developed a multilayer material with lower dielectric constant than standard FR-4 material by employing resins with superior dielectric properties, which were produced based on our proprietary resin modification technology.

$$Zo = \frac{60}{\sqrt{Dk}} \times \ln\left(\frac{4H}{0.67\pi W\left(0.8+\frac{t}{W}\right)}\right)$$ ……Equation to calculate impedance (Stripline)




| Item | Unit | Dk = 3.5 | Dk = 4.3 |
|---|---|---|---|
| Impedance (Zo) | Ω | 50 | 50 |
| Layer thickness (H) | μm | 130 | 130 |
| Line thickness (t) | μm | 12 | 12 |
| Line width (W) | μm | 52 | 40 |

Figure 1 Effect of using low dielectric constant material on PWB design

## 4 Technical Content

### 1. Design concept of MCL-E-78G

MCL-E-78G adopts a resin with superior dielectric properties, which was produced based on our proprietary resin modification technology. It uses thermosetting materials with less hydroxyl group generation; hydroxyl group is the cause of dielectric properties declining, thus allowing both superior dielectric properties and high heat resistance to be attained. We also used halogen-free flame retardants with high thermal-decomposition temperature, Tg and superior dielectric properties.

### 2. General characteristics & properties of MCL-E-78G

Table 1 shows the general characteristics & properties of MCL-E-78G. It has dielectric constant (Dk) 3.5 and dissipation factor (Df) 0.011, which outperform the equivalent values for standard FR-4 material. Its superior heat resistance is also in evidence, having 160°C or higher Tg by the TMA method, 60 minutes or longer T-288 by (IPC TM-650), and the thermal-decomposition temperature of 380°C or higher (5% weight loss). It also achieves excellent reliability, as there was no insulation deterioration, even after 1,000 hours of the CAF resistance test (between through-hole walls: 0.3 mm, 85°C/85%RH, and applied voltage: 100 V).

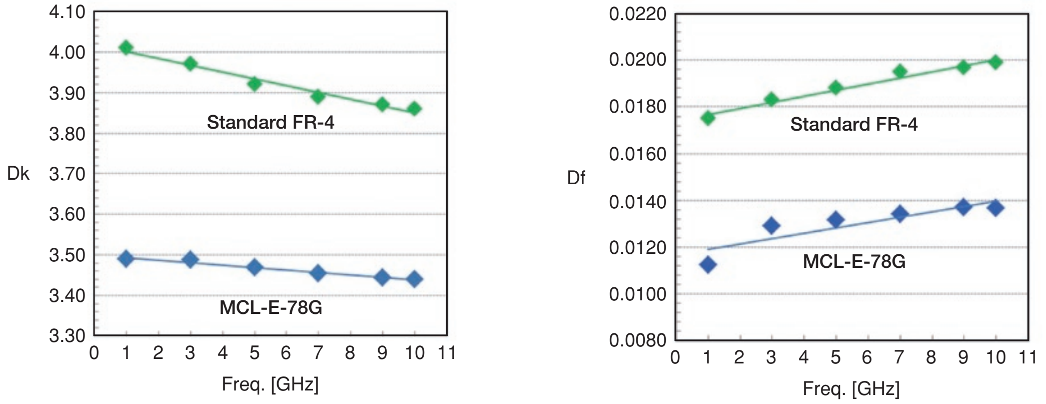
Table 1 Properties of MCL-E-78G (thickness 0.8 mm)

| Item | | Unit | MCL-E-78G | Standard FR-4 |
|---|---|---|---|---|
| Flame Retardant | | — | Halogen-free | Halogen-free |
| Dk (1 GHz) *1 | R.C.=70% | — | 3.4-3.6 | 3.9-4.1 |
| Df (1 GHz) *1 | R.C.=70% | — | 0.009-0.012 | 0.016-0.019 |
| Decomposition temp. (5% wt loss) | | ℃ | 380-400 | 380-390 |
| Tg | TMA | ℃ | 160-170 | 155-170 |
| | DMA | ℃ | 200-220 | 195-215 |
| CTE | X < Tg | ppm/℃ | 13-15 | 12-15 |
| | Y < Tg | ppm/℃ | 15-17 | 14-17 |
| | Z < Tg | ppm/℃ | 35-45 | 30-40 |
| | Z > Tg | ppm/℃ | 180-230 | 180-240 |
| Cu Peel strength | Outer layer 18 μm | kN/m | 1.0-1.2 | 1.2-1.5 |
| T-288 (w/Cu) | TMA | min | >60 | >60 |
| Flammability | UL-94 | — | V-0 | V-0 |
| CAF properties *2 | 85℃/85%RH, DC100 V | hrs. | >1000 | >1000 |

＊1) Measured by a Triplate-Line Resonator
＊2) Drill bit: Φ0.4 mm, T/H wall distance: 0.3 mm, Pre-condition: Reflow x 2 (Max 265℃)

### 3. Dielectric properties of the MCL-E-78G

Figure 2 shows the frequency dependence of Dk and Df up to 10 GHz. It shows MCL-E-78G has superior dielectric properties as Dk by approx. 0.5 and Df by approx. 0.006 were improved compare with standard FR-4 materials.




＊) Measured by a Triplate-Line Resonator

Figure 2 Frequency dependency of relative dielectric constant and dissipation factor

## 5 Future Business Development

・Development of low transmission loss multilayer materials for next-generation mobile products

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Hitachi Chemical
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# New High Heat Resistant White Molding Compound for LED

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## 1 Abstract

In the spreading LED market in future, it is assumed that LED packages will be used at higher temperatures due to the output design and external circumstances, whereupon the structural materials will require higher heat resistance against discoloration than current situation. The discoloration of structural materials by heat causes to decrease optical intensity of LED output.

To meet these requirements from market requirements, we developed an epoxy resin by introducing a functional group capable of suppressing the discoloration due to heat. Moreover, we finally obtained a new product of high heat-resistant white molding compound materials for LED in combination with a highly reflective filler.

## 2 Key Features

・High heat-resistant white molding material with excellent transfer-molding processability.

## 3 Development Background

The LED (Light-Emitting Diode) is a device used to convert electricity to light energy. Various package structures have been developed for various applications, and in recent years, the 0.5-watt input power class surface mount LED packages have become increasingly popular; mainly for backlight liquid crystal displays and other generic illuminations. Its development trend to date has been to focus on achieving higher efficiency, i.e. packages with higher luminous flux, miniaturization, those allowing higher electric currents to pass through[1], and manufacturing LED packages operable under high-temperature environments with an extended service life. Accordingly, we developed and started selling white molding material for white LED reflectors[2].

However, as LEDs penetrate the market more and more, LED packages will predictably have to withstand even higher temperatures, based on the power output design and operating environment. With this in mind, the structural materials used for their packages should be LED packages subject to even more stringent requirements to operate within a high-temperature environment. Heat resistance means resistance to discoloration and degradation and heat-related discoloration of component parts reduces the light output power emitted by LED packages. To start this product development, we reviewed the molecular design of base resin and developed a new high heat-resistant resin for LEDs, utilizing the key features of epoxy resin to meet these performance requirements for the markets.

## 4 Product Design

### 4.1 Application package

This product is a material related to the surface mount LED package, the latter of which comprises the components shown in Figure 1. These include metals such as lead frames playing the role of electrode and substrate, reflectors as insulation material between electrodes and reflecting light emitted by devices, and a protective device in transparent resin and containing dispersed fluorescent particles to convert wavelength. The developed material applies to reflector material.

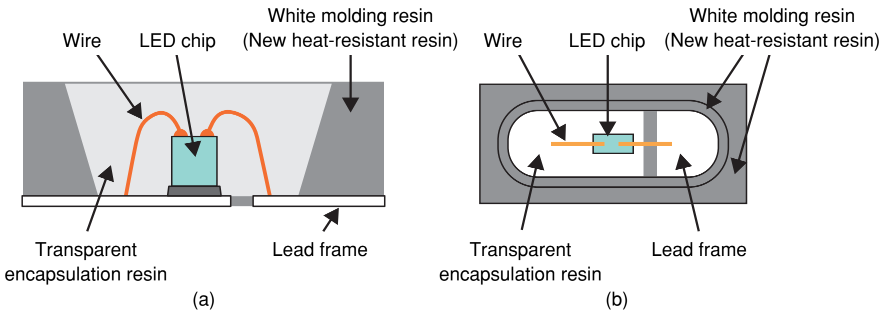


Figure 1 LED package design a) Cross-section b) Top view

### 4.2 Basic Design

Our basic product design centers on composite material; comprising our unique epoxy resin, which is compatible with transfer-molding and package-assembling processes as well as meeting the required performances in terms of optical properties and reliability[3] for LED packages, and highly reflective fillers. A functional group capable of suppressing thermal discoloration was also introduced into the epoxy resin to boost its heat resistance. Thermal discolorations of reflector material are basically caused by heat treatment steps during the processes of assembling LED packages, soldering components by reflow onto a substrate, and heating emitted by LED devices in operation. We confirmed our product could effectively suppress thermal discoloration. Figure 2 shows the reflow- and heat-resistance performances respectively.

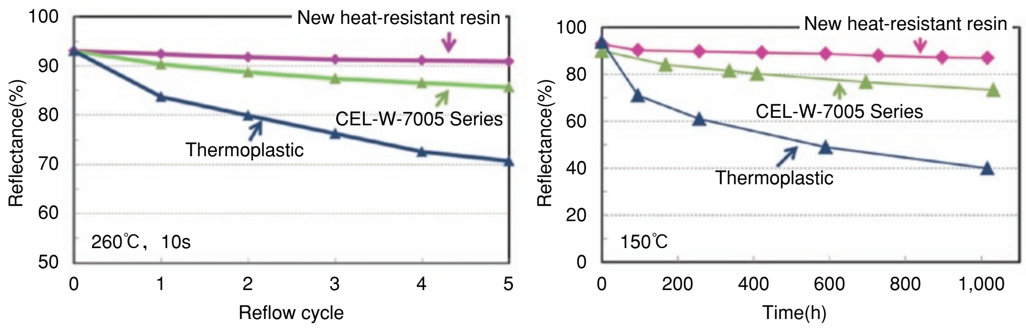


Figure 2 Properties of the newly developed white molding material a) Reflow Resistance, b) Heat Resistance

## 5 Future Business Development

- Sales promotion of newly developed materials
- Take actions in response to enlarged substrate
- Expand product applications


【References】

1) Yukihiro Demukai, Development of LED-Package for Lighting Applications, TOYODA GOSEI TECHNICAL REVIEW, Vol. 53, pp. 22-29 (2011).

2) Hitachi Chemical Co. Ltd., News Release, Expanded Applications of “White Epoxy Molding Resin” for LED-Package (October 13, 2011).

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# Development of Heat-resisted Polymer for Magnetic Powder Coating

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## 1 Abstract

Recently, soft magnetic composites (SMC) have been developed as magnetic parts for high performance motors and actuators utilized in the wide field of automotive and industry. Previously, we developed several resins and inorganic materials[1-2] as binders for SMC which can make them heat-resistant. Furthermore, these kinds of binder materials will be widely used in near future, not only for soft magnetic materials, SMC and bonded magnet, but also for hard magnetic materials used under tougher conditions.

Accordingly, it will be needed for binder resins of magnetic resins that the functional properties should be improved in heat- and weather-resistance than ever before.

In this report, we describe the further development of the heat-resistant binder resin acquired in our development of epoxy molding compounds for semiconductor packages, which can give the optimized properties, especially higher mechanical strength of green compacts, even under higher temperature more than 150°C.

## 2 Key Product Features

- Significantly improved heat resistance of cured resins by optimizing the compounding ratio of thermoset resins, and the selecting the hardening accelerator.
- Molded and cured magnetic core made of magnetic powder coated with our binder resins showed superior mechanical strength in the range of higher-temperature more than 150°C.

## 3 Development Background

For many years, we have been engaged in developing solid epoxy encapsulation materials for electronics components and in-house technologies for curing behavior and cured products, which were accumulated during their development processes. By replacing fillers with magnetic powders, we assumed that these technologies in their current form could be applicable to binder materials capable of boosting magnetic component performance.

In general, resin-bonded magnets include injection-molded magnets, which show superior productivity and corrosion resistance and in which Nylon and PPS [Polyphenylenesulfide] are often used as binder resins for them. However, issues of compressibility and dimensional stability remain pending. Conversely, compression-molded magnets, which show excellent compressibility and magnetic property, are drawing attention under current circumstances amid soaring demand for high performance magnets. Widely used and well-known, conventional, epoxy resins have often been used as binder resins for manufacturing of compression-molded magnets, but room for improvement remained in various aspects of performance, including dimensional stability, heat resistance and corrosion resistance. Developing compression-molded magnets at our company could be traced back to our developmental work on material compositions as we were in a position to be able to develop binder resin coating technology for magnetic powder and molding technology comprehensively. In this development, we engaged in a thorough examination of traditional binder resins and finally developed a new resin composition “HC-Re01”, which shows no decline in mechanical strength, even at a temperature range beyond 150°C and good operability, by taking into account material performance issues derived from tests relevant to operability during coating, cured condition and thermal decomposition behavior.

There are a few performance requirements for binder resin coated on the surface of magnetic powder, including no unwanted progression of curing reactions within the powder handling temperature range, effective mold-releasing characteristics after the compression molding of magnetic powder, short curing time of green compacts and cured them with high mechanical strength.

We designed the composition of epoxy-based thermoset resins to meet these performance characteristics as mentioned above, the design concept for which is indicated as follows:

## 4 Product Design

**Binder resins for cured green compacts with high mechanical strength**

To achieve cured green compact with sufficient strength (as crushing strength), even at temperatures of 150°C or more, cured epoxy resins with a higher glass transition temperature will be required. To do so, the greater the branching degree and the more functional groups exist on a molecular level (i.e. the more reactive sites per molecule), the more favorable the selection of epoxy resin and hardener (curing resin) becomes. If so, when these resins react sufficiently on a molecular level, they can give highly cross-linked cured green compacts to generate mechanically strong them. It is also important for these resins to rapidly kick-start reactions in the curing process (heating step) and complete the curing reactions to the full extent. In fact, even if the resin composition is optimized but the cross-linking reaction has not progressed sufficiently, a cured green compact of sufficient expected strength cannot be produced due to incomplete cross-linking. In other words, to have a complete curing process (cross-linking reaction), the resin system should be kept unreactive until just before starting the curing process but rapidly activated once the curing process (heating step) started. Therefore, we thought that using hardening accelerators with so-called "thermal latent generation (a performance property to rapidly facilitate reactions above a certain temperature)" may be effective. Figure 1 shows schematic drawings of the molecular design concept for binder resins which we developed, while Figure 2 displays mechanical strength performance graphs of cured green compacts of magnetic powders applied to new binder resin.

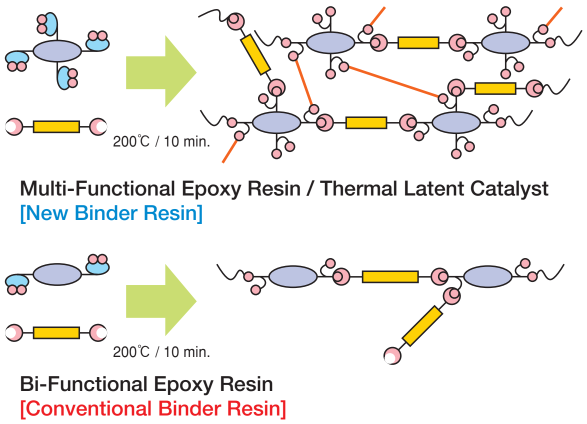


Figure 1 Designing of binder resin on the molecular level

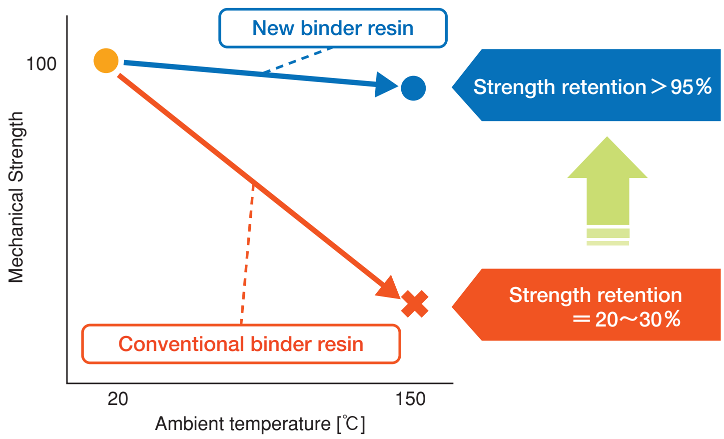


Figure 2 Strength property of cured green compact applied to new binder resin

As shown in Figure 2, the mechanical strength of the cured green compacts of magnetic powders using a new resin composition HC-Re01 is sufficiently retained in the high-temperature zone (near 150°C) where the cured green compact using conventional resin products would have rapidly lost mechanical strength. It was therefore verified that the cured green compacts of magnetic powders applied to new binder resin composite, HC-Re01, can show sufficient mechanical strength stability, even if used as magnetic components within a high-temperature environment.

## 5 Future Business Development

- Study on the applicability of binder resins of various magnetic powders.
- Sales expansion of various stators and rotor cores using our new binder resin, which is operable within a high temperature range.

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# Silk Fibroin Sponge Sheet for Skin Care

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## 1 Abstract

Silk yarn has been used as fabric on account of inimitable shininess and texture, and also as surgical suture on account of high strength and bio-compatibility. By using fibroin protein that is the main constituent of silk, the utilization as the film, the powder, and the sponge is considered[1]. There were some reports how to form the sponge, but the strength of the sponge was insufficient. Therefore, the sponge has not been implemented yet[2]. We have examined how to make the high-strength sponge, and have succeeded in getting the sheet form of the high-strength sponge using the fibroin protein[3]. We propose our sponge sheet as skin care materials, because our developed sponge sheet maintains the good feeling of silk itself, and has the high water absorbency, the high water holding property, and the high adherence.

## 2 Key Points of the Development Product

- This development product comprising natural silk fibroin offers a uniquely soft and tender touch.
- Safety tests, including for cytotoxicity, skin sensitization and human patch were successfully conducted and a high level of biological safety was also confirmed.
- Skin care material with superior performance in terms of water absorption, retention, skin adherence and transparency compared to nonwoven cotton fabric frequently used in items such as face masks can be offered.

## 3 Development Background

We started developing the sponge sheet for skin care by focusing on silk fibroin as a bio-derived material, produced by the silk worm, as part of work to develop life science-related products compatible with changing consumer behavior of recent years in areas such as the environment, safety and health. Figure 1 shows various product forms produced from raw material fibroin. In this report, we specifically focus on sponge among these product forms.

Collagen has been known as a sponge which can be formed from protein but lacks strength as a skin care material. Silk fibroin sponges outperform collagen in strength terms but are not good enough for use as a skin care material. We improved the sponge manufacturing technology developed by the National Institute of Agrobiological Sciences [1], and developed a high-strength sponge which was suitable as a skin care material.

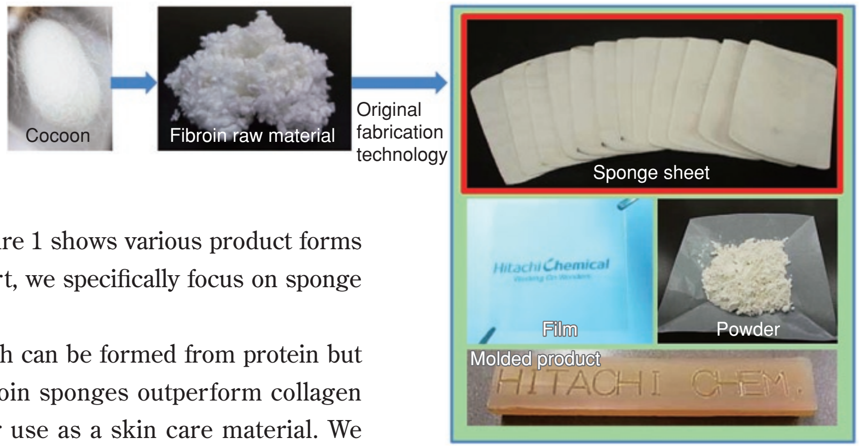


Figure 1 Various fibroin product forms

## 4 Technical Content

Table 1 shows the characteristics of the fibroin sponge sheet, while Figure 2 shows an SEM image of the fibroin sponge sheet. Structural components such as void size and content, and mechanical properties such as tensile modulus and compressive hardness are controllable at will within the ranges described in Table 1.

The characteristics required for skin care material were evaluated against cotton spun-lace nonwoven fabric (hereinafter referred to as nonwoven fabric), which is often used for face mask material, as a control material. Under microscopic observations of sponge sheet and nonwoven fabrics, the surface of the sponge sheet looks smooth without fluff in comparison to nonwoven fabric, which means the sponge sheet can offer a tender touch due to differences in surface structure. Figure 3 shows the water

absorption rate per own weight after water was absorbed in the sponge sheet and nonwoven fabric for 5 minutes. The sponge sheet absorbed approx. 15 times its weight in water at an absorption rate approx. double that of nonwoven fabric. We assume that this difference was attributable to material and structural differences. Figure 4 shows the measurement results of the stress required for releasing wet sponge sheets and nonwoven fabric after they touched the skin. It shows that the adherence strength of sponge sheets is higher than nonwoven fabric by approx. 1.8 times. This result also shows that sponge sheets have better concave/convex skin surface tracking capability than nonwoven fabrics. Figure 5 shows the measurement results of the water dripping ratio when a wet sponge sheet and nonwoven fabric are held in the air for 30 seconds. These results indicate scope to comfortably wear a wet sponge sheet face mask without dripping skin lotions, for example, when a sponge sheet is used as a face mask base material. Figure 6 shows the color differences between a wet sponge sheet vs. the standard Japanese skin color plate and wet nonwoven fabric vs. standard Japanese skin color plate, while both the wet sponge sheet and wet nonwoven fabric are placed on the standard Japanese skin color plate. The smaller the color difference, the more transparent it appears. The greater the color difference, the more opaque it becomes. In conclusion, this result indicates that the wet sponge sheet can deliver a more transparent feeling than wet nonwoven fabric, and can reduce feelings of discomfort while wearing a wet face mask.

Table 1 Characteristics of the fibroin sponge sheet

| Item | Characteristics |
|---|---|
| 25%Compressive hardness | 2-400 kPa |
| Tensile modulus | 0.01 kPa-10 MPa |
| Void size | 1-300 μm |
| Void content | 50-98% by volume |
| Heat resistance | 150℃ or more |
| Solvent resistance | Stable |

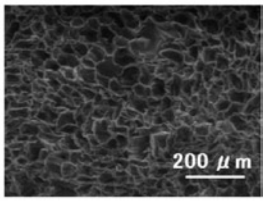


Figure 2 SEM image of the fibroin sponge sheet

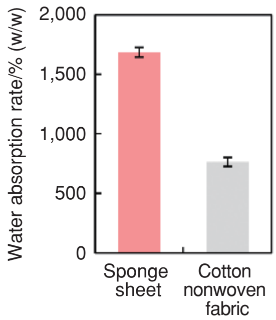


Figure 3 Comparison of the water absorption ratio between the fibroin sponge sheet and the nonwoven cotton fabric

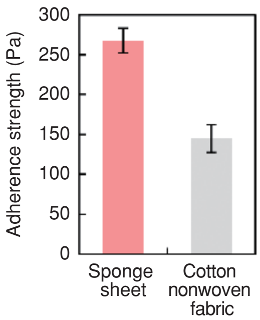


Figure 4 Comparison of the adherence strength to the skin between the fibroin sponge sheet and the nonwoven cotton fabric

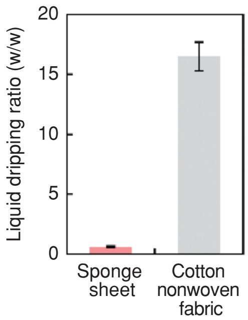


Figure 5 Comparison of the liquid sagging ratio between the fibroin sponge sheet and the nonwoven cotton fabric

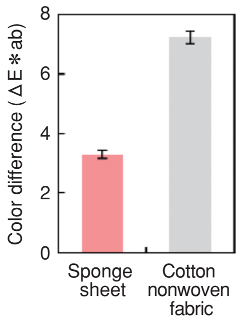


Figure 6 Comparison of the color difference between the fibroin sponge sheet and the nonwoven cotton fabric, compared to the skin color

Figure 7 shows a sponge sheet punched in a face mask sheet. Since any kind of shape can be easily punched out with the Thomson blade, the sponge sheet has various uses.

Figure 7 Facemask made from the fibroin sponge sheet

## 5 Future Business Development

Applications for medical use, including wound dressing, hemostatic material, sustained release dosage form and tissue engineering scaffold material.

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3) Naosuke Sumi and four other colleagues: Proceedings of the Lecture about the 18th Polymer Materials Forum, The Society of Polymer Science, Japan, 2009, p. 201.

## Editor's Note

We have a vision of creating "new value" beyond the field of chemicals by rising to the challenge, stepping into the unknown world of science and eliciting "surprises" that exceed the expectations of society and customers. In this context, the central theme of this report, namely to build businesses related to "Environment & Energy" and "Life Science", remains an indispensable part toward realizing our vision and we hope you will gain an insight into our business activities with this in mind.

In the "Environment & Energy" field in particular, we aim not only to promote research and development into four electric energy storage devices, such as lead-acid batteries, lithium-ion batteries, lithium-ion capacitors and capacitors, and organic/inorganic materials used for these devices, and the operational mechanism and technique to analyze causes of any loss of performance in these devices, but also promote a consolidated business plan that transcends the field of chemicals, including a hybrid system that combines the aforementioned four electrical energy storage devices as required, its control system and maintenance services. We will also further push forward with technological developments that will boost the effective use of renewable energies such as mega-solar and wind energy.

We hope the outcome of our efforts to realize our established corporate philosophy, "to contribute to society through technical advancement and products that can open up a new era to help realize a sustainable society", will help create sustainable stability and affluence on a global scale, however slightly.

SU

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