Development of clay-based-film

— A *Full Research* scenario from a viewpoint of encounter —

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An example of *Full Research* in the development of clay-based-film is introduced. Clay is eco-friendly, and it exists abundantly in Japan. Clay-based-films can be used as heat resistant gas barrier materials that in turn contribute to the development of a sustainable industry. Details of the technical development, public information, intellectual property, and technology transfer occurring during the process from the initial invention to practical application are discussed. At the same time, the interactions of people and research groups related to the development process are analyzed. In addition, the effectiveness of a consortium is discussed in the context of an innovation model based on development integration.

Keywords : Clay, *Full Research*, *Type 1 Basic Research*, *Type 2 Basic Research*, practical use research, encounter, consortium

1 Film made from clay

Most conventional gas barrier films were manufactured using plastics. Their gas barrier performances were not perfect, and the researches were conducted for “clay-plastic nanocomposite material” in which clay and other materials were added as fillers to enhance heat resistance and gas barrier property. Gas barrier performance was improved noticeably by adding small amount of clay. From “reverse ideation” that gas barrier property and heat resistance may increase dramatically if the clay used as filler was used as main material rather than as additive, in 2003 we started the development of heat resistant gas barrier film composed of clay[1]-[3].

Clay crystal is thin plate about 1 nm (1/1,000,000 millimeter) thick. “Claist” is a clay film formed to thickness enough to be handled by layering several tens of thousands of thin crystals. Claist can be bent and has high gas barrier property against oxygen and hydrogen gases under high temperature condition. It is made by cast method, which is a simple method where clay dispersed in liquid is dried in a tray, and the dried precipitate is peeled off. We conducted film preparation using various types of clays to study their film-formability. We found that clay called “smectite” that readily formed water gel had excellent film-formability[4]. About 30 to 70 % of smectite is naturally contained in mineral called bentonite. Bentonite itself is used as moulding agent of casting sand, drilling mud in construction sites, and impermeable layer of dam and waste disposal site. About 450,000 ton/year are produced in Japan, and this is almost equivalent to the production volume of virgin PET by Japanese companies. More than half of this amount is produced from mines in the Tohoku region. Separation and refining of smectite from bentonite is done by sedimentation, which is a method where the material is dispersed in water and the fraction that does not settle is heated and dried.

That the clay forms film is not a new finding for those of us who study clay. When analyzing crystal structure of clay by x-ray diffraction, the usual method is to use oriented sample where clay-dispersed liquid is cast on glass plate[5]. Since the clay film cannot be peeled off the glass plate, it is not self-standing film[Term 1]. Professor Ernest Hauser of MIT reported self-standing clay film in 1938[6]. Intended use was wrapping material or alternative to paper. Although the potential of clay as film material was indicated, it seemed not to be commercialized as product. It may be because no functional or economic advantage could be discerned against competitive material paper.

Seventy years have passed, and there are now many products that require gas barrier such as food packages and electric products. Moreover, special needs arose for containing hydrogen at high pressure while maintaining lightweight as system to be installed in vehicles such as rockets, aircrafts, hydrogen vehicles, and fuel cell vehicles. In petrochemical plants, leak must be controlled as much as possible to reduce volatile organic compound gases. New materials using clay were deployed to meet such new demands.

2 Invention of clay-based film and establishment of the concept

2.1 Discovery of gas barrier property of clay-based film

The Author was initially studying clay compaction to assess artificial barrier in waste disposal sites[7]. Permeation of water through clay compaction was extremely slow, and it required long time for measurement. In some cases, maximum 500 days were necessary for measurement. To complete the measurements in short time, we devised ways of thinning

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the compaction that was about 16 mm thick. Although measurement time was shortened using the thin material, the uniformity of the film greatly affected the measurement precision. We repeatedly formed film on filter paper to achieve uniform thickness. At this stage, we were working on clay film with focus on its function as water barrier. The finding of this project was reported but did not attract much attention.

AIST Tohoku was established in 2001, and research unit "Research Center for Compact Chemical Process" was established in 2004. There, I presented this film to my supervisor who commented that it could be used as sealing material of micro-reactor using hydrogen gas, and I started research of clay film as gas barrier material.

Self-standing ceramic film had numerous small cracks through which air molecules could pass. Therefore, high gas barrier property could not be expected, and ceramic film has not been considered for gas barrier. Clay film certainly showed high barrier property against water, but that was because clay absorbed water, expanded, and filled the cracks. Therefore, people who made ceramic films never considered using them as gas barrier material. The idea that clay film might be used as gas barrier material was probably gained from the fact that the appearance resembled the sealing material called Teflon tape. Teflon tape was actually used as seal in micro-reactor, but it can not withstand over 250 ºC. Efforts were continued on clay film to improve strength and flexibility like Teflon tape, and to reduce pores that resulted from air bubbles. The idea for use was born first from the appearance of the material, and then expanded through human five senses such as sight and touch.

2.2 Demonstration of gas barrier property of clay-based film

While clay film lacked sufficient strength initially, improvements were repeated based on discussions with my supervisor, and in few months, we had a material that could be used as seal in micro-reactor. In the micro-reactor trial, hydrogen had to be sealed off up to about 300 ºC, and no conventional sealing material could achieve this condition. Upon assessment, sealing property achieved to be good. In principle, it was thought that clay did not hinder permeation of hydrogen, but it was unknown whether the sealing property against hydrogen would be lost by addition of binder. Later, using the tuturous model[8], it was found that high gas barrier property was obtained when clay was excessive. Specifically, gas barrier property of 1,000 times higher than the binder material could be obtained when the clay weight ratio was 94 %. The high barrier property according to the principle was experimentally demonstrated, and the basic concept of clay film was established[9].

3 Application development of clay-based film

3.1 Analysis of demand

Press release for clay-based film was issued on August 11, 2004. The highlight of the release was heat resistance of maximum 1000 ºC and gas barrier property lower than detection limit. We also frequently participated in exhibitions, and also wrote several technical reports in journals.

As result of concentrated publicity in short time period, there were over 300 inquiries, and we accepted technological consultations with about 150 companies. It was found that this material held potential for diverse usages including heat resistant flexible film, display film[10][11], graphite composite material, electromagnetic shielding material, condenser seal, packaging material (high barrier paper container, high barrier flexible package), and hydrogen sealing material.

3.2 Enhancement of intellectual property rights

Looking at the timing of disclosure of patent application, presentation at academic societies, and publication in journals, we took measures to enhance the patent in the council that discuss patent enhancement among the Intellectual Property Department, AIST Innovations, and research units. Specifically, preceded technological surveys were conducted in August 2004, December 2004, and February 2005, and enhancements of intellectual property were intensively conducted by Intellectual Property Strategic Enhancement Team (organization composed of smaller number of people than Patent Enhancement Council, to enhance certain intellectual property) in December 2004, June 2005, March 2006, May 2006, and September 2006. Selections were made on fields and R&D topics that should be developed internally within AIST, and fields and R&D topics that should be developed jointly with private corporations. Specifically, we decided to concentrate our application on material patent, part of manufacturing patent, and part of application patent that could be developed in AIST. Patents that were applied for application included flexible substrate, solar cell, and materials for fuel cell. These were areas to which clay-based films were provided within AIST and good results were obtained in preliminary tests. Of course, some did not go well and applications were not submitted. During this period, about 40 patent applications were applied. Attainment of sufficient quality and quantity of the intellectual properties and application for patent individually by AIST for this technology started in 2003 and reached peak in 2004-5.

3.3 Policies for technological transfer and joint research

Technological transfer was conducted through research sample provision contract and research information disclosure contract. There were about 70 former cases and about 20 latter cases. In technological transfer, we worked to accumulate sufficient manufacturing know-how and to communicate this know-how accurately and in detail. In research information disclosure, we actively accepted observation inside the lab as well as disclosure of know-how book and presentation of undisclosed patents. Disclosure of undisclosed patents and know-how books were not very detailed, and the companies often could not reproduce the results completely with that information only. As result of lab observation, most companies were able to successfully create film with quality equivalent or higher than the AIST sample, and this style of research information disclosure proved to be effective in transferring technology.

In 2004, we commenced joint research by different usages based on research information disclosure contract. Such joint researches increased around 2005, and the development stage of clay-based film gradually shifted to Applications Research. Even in case the film would be used in same product, if division could be made between upstream and downstream, we made it possible to start joint research. It was thought that as the R&D stage progressed, vertical collaboration from upstream to downstream companies would accelerate R&D. At the same time, there was possibility of conflict of interests, and it was necessary to take measures to maintain friendly development relationship. The employees of AIST could not be involved in details due to nondisclosure obligation, and it was useful to have a consortium composed of major research members. The Research Center for Compact Chemical Process is an organizer of Green Process Consortium (GIC), and supports horizontal and vertical collaborations among companies.

The number of joint patent applications with companies increased from 2006, and in 2007, 80 % of all patents filed were joint applications. One characteristic of clay-based film development is that there are more than 10 companies with which joint applications are filed.

4 Development of asbestos-substitute gasket

Among the companies from which we received technological consultation, we decided to develop sealing material for use under high-temperature condition with Company J in the Project to Support Regional Small & Medium Companies, Ministry of Economy, Trade, and Industry in FY 2005. Based on the results obtained, we developed gasket product with heat resistance, durability, and chemical resistance superior to those of existing non-asbestos products, as well has excellent manageability equivalent to asbestos products, by compositing expanded graphite and heat-resistant clay-based film, as project of New Energy and Industrial Technology Development Organization (NEDO). The newly developed gasket can be used widely in chemical plants such as oil refinery and thermal power plant.

4.1 Problems in conventional asbestos-substitute gasket

In many chemical industries, gaskets are used to prevent leakage of liquid or gas in the pipe joints in the production process under high temperature condition. Asbestos products were used widely in high temperature area. While immediate measures had to be taken against asbestos health hazard, development of alternatives was in progress and assessment of safety and reliability were not sufficient. Expanded graphite gasket had many advantages such as excellent sealing, long-term storage, and ease of handling, and was highly regarded as non-asbestos product. However, bonding among graphite powders was not strong, and there were problems such as “powdering” where powders flaked off from the product surface, and “adhesion” where the graphite burned on to the metal surface where the gasket came into contact. Moreover, in oxygen atmosphere and temperature of 400 ºC or higher, oxidation and deterioration progressed and the gasket wasted away, thus loosing sealing property.

4.2 Scenario setting

4.2.1 Improvement of heat resistance of gasket by compositing expanded graphite and clay

It is difficult to solve the phenomenon where graphite burns in presence of oxygen at 400 ºC or higher. However, clay is oxide and has high heat resistance. Clay used in clay-based film is stable to about 600 ºC, and we considered improving the overall heat resistance by mixing and compositing clay. Since clay-based film has high impermeability against oxygen, we thought it was possible to lengthen the lifespan of gasket by slowing down transfer of oxygen by coating with clay-based film.

4.2.2 Prevention of powdering and burn-on by addition of clay layer

Powdering of expanded graphite could be solved by applying an uniform coat of clay-based film on the surface as in the current fluororesin products. Burn-on was thought preventable by isolating metal surface of the flange from graphite by clay-based film coating.

4.2.3 Improvement of sealing performance by flattening the gasket surface

It was possible to reduce the amount of liquid that leaked between the expanded graphite gasket and metal flange by flattening the surface of clay-based film used to coat the gasket.

4.3 Elemental technology

4.3.1 Nanocompositing technology

Clay has higher heat resistance than plastic. Also it expresses gas barrier property by fine preparation. However, there were three major issues in using clay film as gas barrier material. One was existence of cracks. It was not easy to completely remove small cracks through which gas molecules could pass even though clay-only film appeared uniform. In gas barrier material, just one crack may cause performance deterioration. Second was low mechanical strength. Although clay film could be bent, it was not flexible like plastic. The film was weak, and once a crack formed it could be easily broken. Third was weakness against water. As mentioned earlier, the condition of clay to be used in self-standing film makes good aqueous dispersion, but this also meant that clay film dissolved readily in water. It had problem of poor steam barrier property that was often demanded of gas barrier material. Unless these three issues were cleared, there was no future as general-use gas barrier material. It seems that fairly large number of engineers gave up because they were unable to solve these issues.

To solve these issues, nanocomposite technology was applied. Clay raw material and organic compound that would act as binder were uniformly mixed at microscopic level to form the film. In many cases, dispersal technology involving pretreatment using the charged state of clay surface was used. By nanocompositing, it was possible to remove the crack on the clay film and to improve mechanical strength and water resistance. While in ordinary nanocompositing, small amount of inorganic material was introduced into organic material, in case of clay-based film, small amounts of organic material were present in the clay, and the composition was opposite.

4.3.2 Clay-based film manufacturing technology

Thousands of sheets were created to obtain the best film-forming method. Ultimately, we made clay film every day for five years, and were able to accumulate the know-how of film forming. As result, we could create clay film of 10 μm thickness with good reproducibility. At the same time we investigated the coating method, and learned that dip coating, spray casting, cast method, and bar coating method could be applied. During the aforementioned research sample transfer contract, we spent effort on quality control even at lab level. The samples were carefully created, reproducibility checked, and main property values were obtained as much as possible by subcontracting to external lab to supply property value chart. We established clear specification of film quality before shipment. Specifically, they included size, level of unevenness of thickness, and level of unevenness where clumps could be observed by naked eyes. Such quality control was useful in accumulating manufacturing know-how of clay-based film.

4.3.3 Clay library

In the process of looking for appropriate clay for film, about 130 clay samples were collected from Japan and overseas. These were natural or synthetic clay, and most were available commercially. It included inexpensive clay samples without purification. The data collection of assessment of film-forming property of these samples is in progress, and clay suitable for clay/expanded graphite composite was selected from this library.

4.4 Integration process

4.4.1 Selection of coating method with good adhesivity to expanded graphite

Heat resistance of at least 400 ºC was required for the gasket. Therefore, organic adhesive agent could not be used. Initially, pressing method was used. Although certain adhesivity was obtained, there was problem of air entering between expanded graphite and clay film. Dip coating had advantage of prepareing coating layer to form on the sides. Upon trials, clay film of about 20 μm thickness could be formed on the expanded graphite surface. Water was used as solvent.

4.4.2 Selection of appropriate combination from various raw materials (clay and additive, clay blend)

We worked on selecting clays from the clay library with excellent adhesivity to the surface of expanded graphite. Since transparency was not demanded in this clay-based film, screening was done mainly for natural clay due to cost concerns. As result, it was found that film forming and adhesive properties were excellent in clays that contained mineral called smectite. Epoxy, phenol, and polyamide resins were selected as additives, and optimal material among them was selected in the final specification. For heat resistance, the amount of additive was minimized enough to maintain mechanical strength. The solid-liquid ratio of coating paste was increased to shorten duration required for drying, and clay blend was investigated to create film with excellent fabrication property. Coating film with excellent film property as well as fabrication property was made using clays with different types.

4.4.3 Feedback system for utilizing the assessment result of element test and actual plant trial

Element test was conducted by gasket manufacturer Company J, and good results were obtained for sealing, sticking, and manageability assessments.

In the product development process, we were able to obtain cooperation of Company M, a user company and a member of GIC. Testing was done by installing a bypass in actual petrochemical plant even though the gasket had no previous record of performance. This vertical collaboration effort was deployed under NEDO Urgent Asbestos-Substitute Development Project, and feedback system was established where element test and assessments in actual plant test could be utilized for improvements. Basic data on safety and reliability was obtained and asbestos-substitute gasket was commercialized for use under high temperature condition in 2007[12]-[14]. A gasket factory constructed and started the operation in Osaka in September 2007, and the product is used in about 40 plants throughout Japan as of July 2008. Asbestos-free plants were realized with the introduction of this gasket. This result was acclaimed highly, and the gasket won the Exellent Award of the 2nd Monodukuri Prize in 2007.

The primary reason this product development was achieved in short time was due to precise idea from the president of Company J who thought the problem of surface powdering and burn-on that were disadvantages of conventional expanded graphite gasket could be solved by potential technology of clay-based film. The second reason was smooth technological transfer; third was relentless effort of the engineers at Company J; fourth was cooperation and decisions of users obtained through GIC; fifth was quick managerial decision of Company J and practice of technological sales throughout Japan; sixth was close network between NEDO and AIST; and seventh reason was the fact that production was possible by single company.

External factor was the goal for total abolishment of asbestos products by 2008, and the market demanded substitute products. This newly developed product can replace about 70 % of the asbestos gasket products, and we plan to conduct further performance assessment tests, as well as improve long-term reliability, and plan to expand into automobile industry and electric industry as well as chemical plant industry. At the same time, we are developing products for use in higher temperature.

As AIST's internal project, original R&D project to produce the patents held by AIST were conducted in FY2006 and 2007. Also, several grant-based joint research are in progress, and we are steadily moving on toward second and third successful developments.

Creating actual cases of product development and accumulating basic and production technologies at this stage are keys to success of development. Mass-produced, small profit products must have balance between supplier's production technology and user's product development research, and certain time is necessary for shaping of the market. On the other hand, product development of gasket was done by single company, and development was accomplished in short time without waiting for formation of corporate collaboration. If there are cases of developments, the desire for product development of researchers, engineers, and managers in other fields increases, and this is another reason we think creating successful cases is very important.

5 Encounter and research development in *Full Research*

Here I shall analyze the encounters in *Full Research* using as example the development process of clay-based film.

Specifically, I shall categorize the encounters in the processes of development of invention and progression to *Full Research*.

In *Type 1 Basic Research* stage, about 70 years have passed since humans invented clay film and about five years since the Author started research on clay-based film. During that time, changes took place in the types of industrial products and demands in performance of materials, and new potentials arose from the old seed technology. The manufacturing know-hows of clay film were accumulated from the basic research of barrier for disposal sites of high-level radioactive wastes and industrial wastes. These basic technologies had been achieved to some level and were available as papers and reports. With the establishment of new research unit, researchers of different backgrounds were able to encounter, and development of sealing material was requested from the Author's supervisor who faced the issue of sealing in micro-reactor[3][15]. There were two points in this encounter. First was the match between potential and demand; second was proposal without preconception.

For the first point, detailed explanation using Figure 1 is provided. For a breakthrough such as discovery or invention to happen through encounter of individuals or groups A and B, at least A should have the desire to seek solution to the problem. Specifically, to achieve highly specialized R&D I, A strongly demands technological development element X that A does not have. B has technology α that is result of R&D II, and α can contribute to X. Contribution of α to X may be close to 100 %, or it may be about 50 %. When A and B encounters, technology for α is presented by B to A. Even when the contribution of X to α is not particularly high, strong desire will push actively search for potential, and potential of contribution of α to X will be found. If the contribution of α to X is high, since the time and resource (people, budget, and facility) for R&D I are available, invention and discovery are achieved in short time. If contribution of α to X is not high, it is necessary to raise the contribution of X by further developing α by feedback to B from A. In this case, cooperative agreement between A and B is necessary. Cooperation may assume the form of joint research III. This stage may be done within AIST or simply as request or order within the research group. At this time, if research II is completed for B's technology α and utilization is being sought, thinking of both parties can match and agreement can be readily obtained. In case research II is in progress, the information may not be disclosed completely and cooperation may be limited.

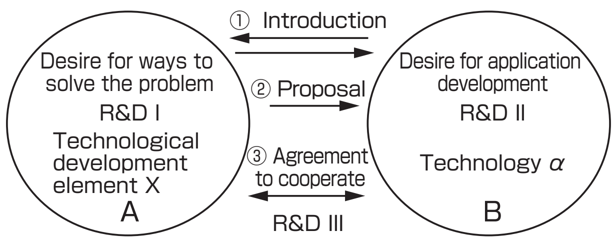

Fig. 1 Encounter in *Full Research* and mutual relationship.

Table 1. Style of encounter in gasket development.

Stage	A	B	X	α	R&D I	R&D II	R&D III	Content of Research	Opportunity for encounter
Type 1 Basic Research	Supervisor	Clay researcher	Hydrogen gas sealing material for micro-reactor	Uniform film using clay-based film	Micro-reactor development	Water barrier research using clay compact	Grant within AIST	Confirmation of high barrier performance, investigation of high barrier performance mechanism	Start-up of new unit
Type 2 Basic Research	Company J	AIST research unit	Improvement of heat resistance of gasket	Heat resistant gas sealing material	Grant within AIST		Support Program for Regional Small and Middle Company, MITI	Development of clay expanded graphite gasket and packing	Technological consultation
Product Development Research	User Company M	Company J + AIST research unit	Asbestos-substitute gasket product	High-performance gasket	Support Program for Regional Small and Middle Company, MITI		NEDO Urgent Asbestos-Substitute Development Project	Development of non-asbestos gasket	Consortium (GIC)
Innovation	User Company Group	Company J + AIST research unit	High-performance gas sealing product	Development technology for high-performance gas sealing product	NEDO Urgent Asbestos-Substitute Development Project		NEDO R&D Project for Industrializing University-Originated Technology	Development of general-use high-performance gas sealing product, data collection for standardization	Product advertisement, etc.

Here, flexible search of potential is expected from A. For example, for technology α, one must use imagination such as thinking about combining with other technologies. It is also necessary to flexibly change the research plan. Next, appropriate explanation for originality is expected from B. Since A does not know technology α very well, it may understand originality wrongly. For the same reason, it is necessary to provide objective advice on whether employment of α is the most appropriate choice among several technologies.

Table 1 is the application of this model to the development of the clay-based film. In *Type 1 Basic Research* stage, clay researcher is B and supervisor is A. In *Type 2 Basic Research* stage, Research Center for Compact Chemical Process is B and Company J is A. In both cases, the encounter is almost ideal. Moreover, in the Product Development Research stage, Research Center for Compact Chemical Process and Company J are B and user Company M is A. The points of this model are: it can be used for analysis for each stage of *Full Research*; and there is optimal timing of encounter. In the process of progressing from *Type 1 Basic Research* to Product Development Research, encounters occur from inside of the organization and spread to outside. Specifically, they spread from highly specialized people who mainly study clay-based film to people who are less specialized, external researchers who have registered ID issued by AIST and to manufacturers and sales people who do not have registered ID issued by AIST.

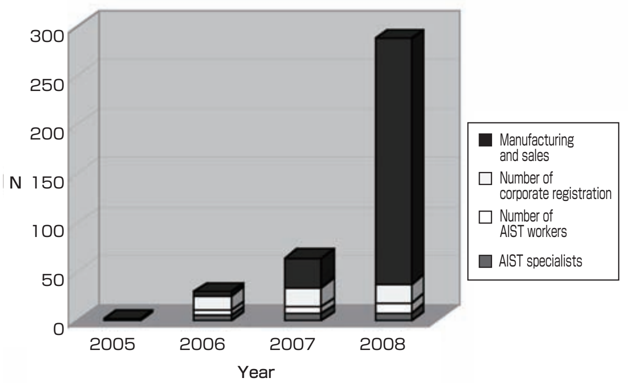

Fig. 2 Expansion of people in clay-based film research (N is number of people involved).

Figure 2 shows the spread of people involved in clay-based film. One of the indexes of evolution of innovation is the fact that the number of people involved increases and population who can benefit as users increases.

6 Integrated development model

Above was the discussion of product development for clay-based film. Product Development Research for clay-based film is divided by products, and these are done with different companies. Also, in some cases, the relationships are upstream and downstream. Specifically, they include companies that produce clay, companies that manufacture clay-based film, and companies that manufacture products using clay-based film. Also, along with vertical relationship as described above, there are companies that share common technological elements, though for different products. For example, they include high water resistance, improvement of transparency of film, or high gas barrier property. To obtain result through balanced development with involvement of all the companies, individual development should shift to accelerated development through information exchange. Since content and progress of joint research with other companies cannot be communicated due to nondisclosure agreement, method for enhancing corporate collaboration through mediation by AIST must be devised, and we are trying to promote collaboration by gradually lowering the walls between the companies using aforementioned GIC. The year 2008, when a industrialization of the product is achieved are produced and joint patents are disclosed, is an appropriate time to start mergeing technologies. In the above model of encounter, the preceding company should be B and the following company should be A in terms of establishment of original technology. AIST does not need to be directly involved in such collaborations.

The multiple development style based on collaboration is called “integrated development,” and is compared to the case of “individual development” where no particular collaboration is done. Figure 3 shows the case where product development A, B, and C of clay-based film are done by integrated development and individual development in solid line and dashed line, respectively. In the case of individual development, development of entire clay-based film is equal to the sum of each product A, B, and C (II). The shift is made to integrated development at time T when product development A is achieved. By providing part of technology and know-how accumulated during the development process of product A, development of product B is accelerated. The technology and know-how accumulated during the development of A and B are utilized in the development of product C. Product development D which could have been only conceived in integrated development may be born. As result, overall development of clay-based film is accelerated (I). In bad scenario, collaboration among corporations may fail, each other's technology cannot be used, and product development may slow down compared to single development (III). In this case, product development may be withheld until the expiration of patent (III).

The case corresponding to D that was born by integrated development includes the development of hydrogen gas barrier material for high-pressure hydrogen gas container[16][17]. This is material where clay-based film is sandwiched between plastic sheets reinforced by carbon fiber, and is in progress with cooperation from manufacturers with potential technology for carbon and clay-based film composite material.

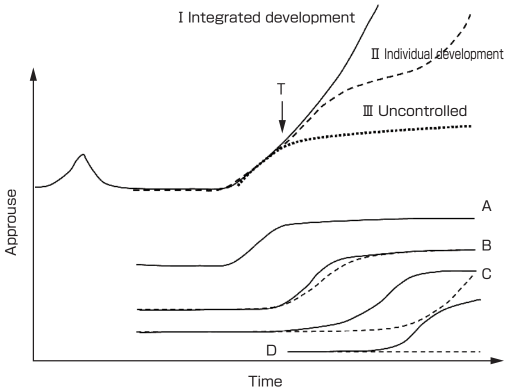

Fig. 3 Concept of integrated development.
A-D are individual product development, solid line is integrated development, and dashed line is individual research.

7 Claist Association

To realize integrated development, we established the Claist Association in August 2008. As mentioned above, GIC, which was a research group for industry-academia-government collaboration at AIST Tohoku, played a major role in the development of Claist, and the Claist Association was created as a subcommittee. As of July 2008, about 30 private companies are members, and we plan to promote practical application of clay-based film while maintaining close collaboration among companies (Fig. 4).

Specific activities include provision of latest technological trends such as patents and papers, management of clay library, operation of Association encounter, and provision of common core technology. Although there are some AIST researchers among the administrator, most are corporate members. That is because it is important to operate the association so it will not interfere with integrated development.

8 Summary

For development of clay-based film, strategic maintenance of intellectual property (quality and quantity) was accomplished through combination of publicity, intellectual property, technological element research, and technology transfer while being strongly schedule-conscious. Manufacturing know-how of clay-based film and roadmap to development were also established.

Encounters at each stage from *Type 1 Basic Research* to Product Development Research were important. Such encounters can be generated by fusion of research groups, publicity, and establishment of a consortium. To incorporate

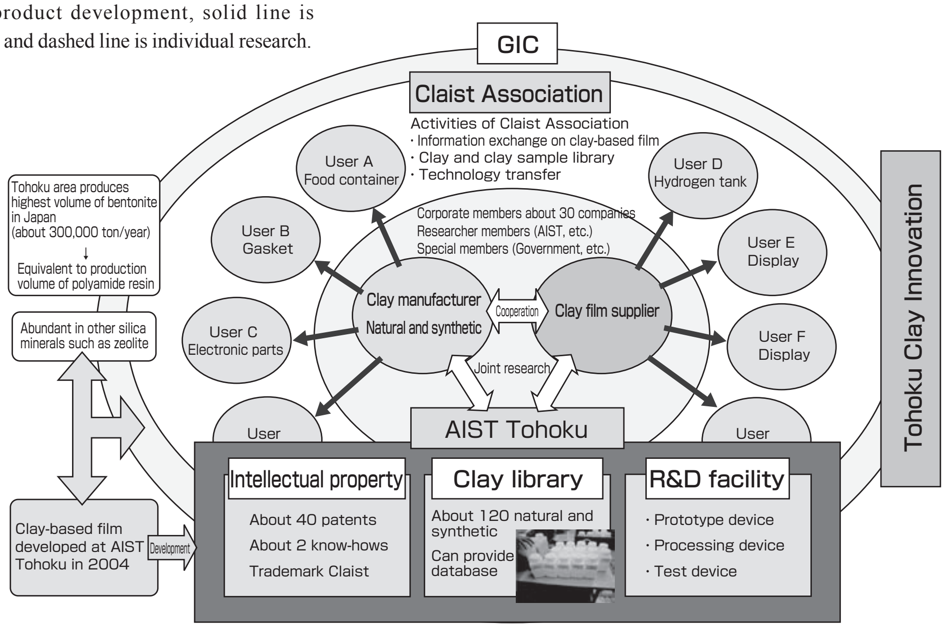

Fig. 4 Mutual relationship between Claist Association and members

these activities strategically into the development process is effective in accelerating *Full Research*. In the case of clay-based film, asbestos-substitute gasket was produced as development case. Through controlled information disclosure, AIST became the center of technology, intellectual property, and information, and took initiative of development. This also means that it has to act as mediator of companies. Consortium activity is an effective way of efficiently mediating and integrating individual product developments.

Finally, by presenting the story chronologically, the latter half of the paper emphasized Product Development Research. However, it is important not to neglect basic research that must be undertaken by public research institution, and it is necessary to maintain manpower for basic research. Currently, we are engaging in basic research to clarify the details of film-forming mechanism of clay and to obtain film flexibility. As result, we are about to generate synthetic clay with film-forming property almost equivalent to natural clay.

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Terminology

Term 1. Self-standing film: Film that can be handled without supporting layer, unlike film that is used to coat a member.

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Discussions with reviewers

1 Overall structure of the paper

Comment (Hiroshi Tateishi)

Since you discussed *Full Research* thoroughly, the focus has become unclear, and the part on *Type 2 Basic Research*, which is the main point of *Synthesiology*, is insufficient. Particularly, I think the first half is merely an "explanation" of the development of Claist as a whole. Since I think the focus of the paper is "the practical application of Claist," I think you should reorganize the structure of the paper.

For example, if "the dream of research" is "the realization of clay-based film and the specific example is the development of an asbestos-substitute gasket," and "the social value of research" is "response to new demand through heat-resistant gas barrier film," I think the corresponding scenario setting, description of elemental technologies, and description of the integration process are necessary. For the paper, you need not to describe all components chronologically, and I think you should sort, select, and discard a bit more.

Answer (Takeo Ebina)

As result of trying to include explanation for all elements of *Full Research*, certainly, the focus has become unclear. I reviewed the structure for the points you indicated. Since "dream of research" is "development of clay-based film and specific example is development of asbestos-substitute gasket," and the "social value of research" is "response to new demand through heat-resistant gas barrier film," I included the corresponding scenario setting, description of elemental technology, and description of the integration process.

2 Addition of process leading to reverse ideation

Comment (Hiroshi Tateishi)

I think the impact will be stronger if you describe how you achieved "reverse ideation" in the discovery of gas barrier property of clay film. In the current explanation, it seems like you just happened to think of it.

Answer (Takeo Ebina)

The discussion on how "reverse ideation" lead to the invention was insufficient. For this point, I described how effort was spent in routine research to achieve invention that other researchers cannot easily attain alone.

3 Mutual relationship of encounter in *Full Research*

Question (Kazuo Igarashi)

In Fig. 1, A is desire for potential and B is desire for application. The two are different phrases but seem to talk about the same thing. Are there differences? Also, in relationship between A and B, it seems that A always takes the initiative, but is this correct understanding?

Answer (Takeo Ebina)

As you mentioned, it does seem that A's desire for potential and B's desire for application seem to talk about same thing. Reconsidering this point, A is desire for ways to solve the problem. Since research is progressing chronologically in B, it can be called desire for application of research result obtained, rather than desire for problem solving. For this point, I changed A to desire for ways to solve the problem.

It seems that in the relationship between A and B, A is always taking the initiative. This description is forced categorization, and I think discussion is necessary of which cases fit this pattern. Yet there are two reasons for setting different positions for A and B. First, I thought this analysis might be useful in consciously creating opportunity for encounter. By making decision on whether one's status is A or B, there will be advantage in how to seek partners efficiently and how to set policy for fruitful encounter. Second, although it is possible to set A and B at same position, in this case discussion from each position cannot be done in the encounter, so it was necessary to give A and B some characteristic. The two who seem to be in same position can be given characteristic as A and B as the content of technology becomes more refined. For example, in one project, there are technological development factors X1 and X2, and technological development element X1 was A's position and factor X2 may be B's position. The two is even in total. Also, I think there are cases where one of them will take initiative in short time, and then shift to joint development.

4 Organization of concept of integrated development

Question (Kazuo Igarashi)

In Fig. 3, it is explained that D can be born as result of integrated development, but seen chronologically, the line starts quite earlier than point T. What is the reason? What does the flat line mean?

In the caption, it is written than D is also individual development, but does this mean thing born from integrated development is developed individually?

Answer (Takeo Ebina)

Figure 3 can be interpreted wrongly as it, and I think it must be corrected so it could be understood readily. As you indicated, D should have the line starting from point T. Solid line is change in case of I individual research, and if this is II integrated research, breakthrough will occur in earlier stage from B to D. I decided to add this line. The cases where the product developments of A, B, C, and D for clay-based film are developed integrated and when they are done individually are represented by solid and dashed lines, respectively.