

New Measurement Technology: Multi-probe System – Toward Direct Measurement of Functions of Nanomaterials and Biomaterials –

TSUYOSHI HASEGAWA (*Affiliated Fellow*) AND KUNIYUKI TADA
Materials and Manufacturing Technology Research Unit

1 Introduction

Progress of many scientific technologies often proceeds together with the development of new measurement technologies. It would not be an exaggeration to say that new apparatus is indispensable for discovering new phenomena and to elucidate them, and this fact has been proven by many researchers who have received the honor of the Nobel Prize. Among the recent Japanese Nobel laureates, the case of Dr. Masatoshi Koshihara (in physics, 2002)^[1], honorable professor of Tokyo University, who has realized the detection of neutrino from a supernova by developing “Super-Kamiokande,” is exactly an example of this fact. Furthermore, development of new measurement technology itself is also being appraised. The case of Mr. Koichi Tanaka (in chemistry, 2002)^[2], fellow of Shimadzu Corp. who has developed the “Desorption Ionization Method” enabling the mass spectrometric analysis of biopolymer, is a direct example. These examples show how important the development of new measurement technologies is for carrying out research work with high originality.

In this report, we would like to introduce the outline and the present status of the development of devices for the “Multi-probe System,” which is expected to contribute to the development of new research areas in the fields of nanotechnology and biotechnology. These fields are considered to be very important for Japan who advocates a technology nation.

In the fields of nanotechnology and biotechnology, development of completely new technologies and products are being sought

by utilizing characteristic functions of newly developed bio-materials and nano-materials. In the research and development with such targets, the first thing to be done is to know the functions of these new materials. The Multi-probe System makes it possible to measure the functions of these new materials, which has been impossible with conventional apparatus, and it is expected that the Multi-probe System will create new methods of research and, consequently, to create new research fields.

2 Outline of the Multi-probe System

The Multi-probe System is a device that uses a plural number of micro probes that directly touch bio-materials or nano-materials to measure functions such as electrical characteristics including signal transmission, and chemical and mechanical characteristics. And the basic technology of the Multi-probe System lies in the scanning probe microscope (SPM)^[3] that uses a single scanning probe.

The SPM is the first device that made it possible to directly observe individual atoms on the surface of a sample, and the use of SPM has rapidly grown after its invention in a wide range of research and development areas. The inventor of SPM was, as in the cases of similar inventions, awarded the Nobel Prize (in physics, 1986)^[4]. Although the Multi-probe System is based on SPM technology, it is a device that enables the direct measurement of functions of nanomaterials and biomaterials, which has never been facilitated by the use of a single probe. As shown schematically in Figure 1, while a single probe provides only

information relating to the area directly under the probe, the use of a plural number of probes enables the obtaining of the total information of the sample including the situations of transmission of signals that are input. In addition, since the System is based on the technology of SPM with a resolution on the atomic level, it is possible to carry out with a spatial resolution on the nano-scale. For this reason, the Multi-probe System is expected to provide new research techniques in the fields of nanotechnology and biotechnology.

The following is an outline of the Multi-probe System and SPM, which is the basic technology of the Multi-probe System.

2.1 Scanning Probe Microscope (SPM)

The SPM, which is the basic technology of the Multi-probe System, is a device that obtains information on the sample surface by scanning the sample surface using a single probe. There are several types of SPMs including Scanning Tunneling Microscope (STM) that uses the tunneling current between the probe and sample for the control of the position of the probe, and Atomic Force Microscope (AFM) that uses the atomic force between the probe and sample for

the control of the position of the probe.

The type of SPM that was first developed by researchers of IBM in 1982 was STM^[5]. When the probe is brought close to a sample within a distance of about 1 nanometer, a tunnel current flows between the probe and sample. The amount of the tunnel current depends on the distance between the probe and sample. For example, if the distance of 1 nanometer between the probe and sample is made shorter by only 0.1 nanometers, the amount of current is increased by a factor of 10. Therefore, it becomes possible to keep the distance between the probe and sample constant by controlling the position of the probe (height) so that the tunnel current is kept constant. If the fluctuation of the tunnel current is kept within a range of several percent, for example, the distance between the probe and sample can be kept constant with an accuracy of 0.001 nanometers (one hundredth of the atom size). When the probe is scanned along the sample surface with such accurate control of distance, the probe would move as if it were exactly tracing the surface of the sample. Thus, the surface topography can be observed by imaging the movement of the probe (see Figure 2). Because the tunneling current is sensitive

Figure 1: Difference in measurement information according to the number of probes

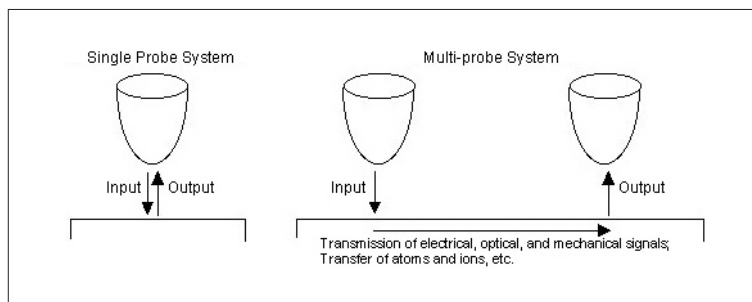
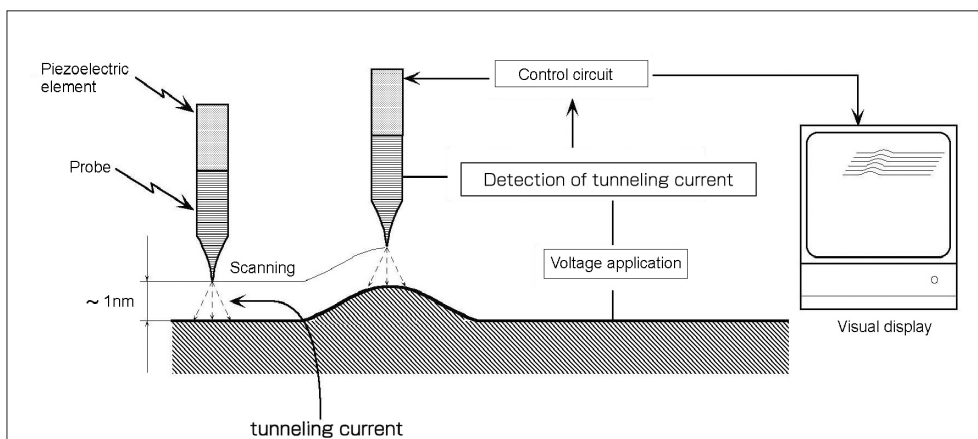


Figure 2: Principle of scanning tunneling microscope



to the change of distance between the probe and sample, individual atoms can be directly observed.

In addition, the STM provides diversified physical information including the energy diagram of electrons derived from current and voltage characteristics between the probe and sample. It can be used not only as a mere instrument for observation but also as a method for fine processing by manipulating atoms, for example, making use of the interaction between the probe and sample^[3]. For this reason, the STM became a device that has been used in a wide range of applications in a very short period of time. It is not an exaggeration to say that the propagation of the STM paved the road to the present day nanotechnology, and the inventor of the STM was awarded the Nobel Prize for this contribution.

Other types of STM include the AFM and the Scanning Near-field Optical Microscope. The AFM is used to control the position of the probe by detecting the weak force acting between the probe and sample (atomic force) using a miniature detector of a cantilever type. And the Scanning Near-field Optical Microscope (SNOM) is a device to control the position of the probe, by detecting the special light that exists only in the near field of the sample using a probe of optical fiber provided with a sharp tip. These microscopes can be used for insulator samples that do not pass the tunneling current, and also for the measurement of various physical values with a nano-scale spatial resolution that cannot be measured with the STM, such as hardness and luminescence properties.

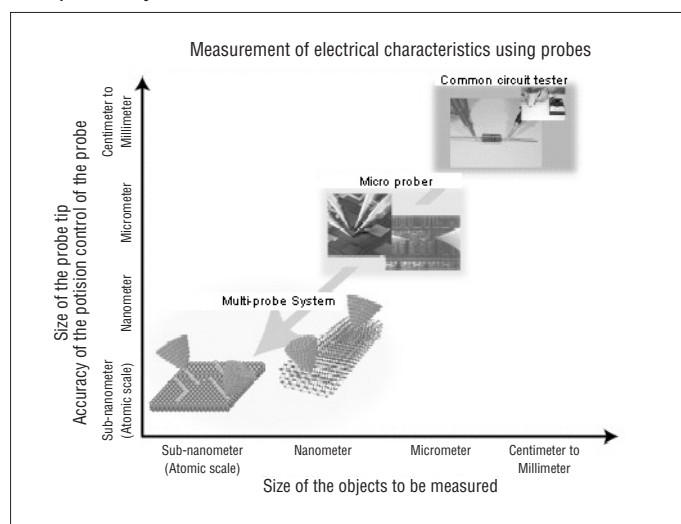
2.2 *Background of the development of the Multi-probe System*

In the development of devices that have functions, it is always necessary to confirm these functions. In the development of semiconductor devices of present day, for example, a device called a prober is used to measure the performance characteristics of transistors. This measurement is performed using a plural number of probes provided with tips on the micron-scale, which are brought to contact to the measuring electrodes of the sub-millimeter order. Therefore,

this measurement can be carried out only on elements for the confirmation of performance characteristics called TEG (Test Element Group). To measure the performance characteristics of individual transistors that are actually used for operation, it is required to bring a plural number of miniature probes to an area of $0.1 \mu\text{m} \times 0.1 \mu\text{m}$ or smaller, and this cannot be done by conventional measuring instruments.

In the development of nano-devices for the future, the size of area to be approached becomes still smaller. In addition, even the technologies for wiring have not been developed for many cases of the development of nano-devices that use new materials such as carbon nanotubes and DNAs, and there is no other means than the direct contact of the probe with the sample to be measured. In order to solve this problem, a Multi-probe System that uses several SPM probes that can approach areas on the nano-scale is being studied and some researchers are already carrying out basic experiments. Since direct contact between the probe and sample is required for the measurement of electrical characteristics, the development of nano-contact technology that applies the probe control technique using the SPM with an accuracy on the nano-scale is also being carried out. The Multi-probe System is also able to elucidate various phenomena such as antibody antigen response and ion transmission with a high spatial resolution, and it is expected to play an important role in medical applications.

From a mechanical point of view, the Multi-probe System is a device equipped with a plural number of SPMs. However, it requires the development of new techniques that have never been used for conventional equipment, such as the control of relative positions of a plural number of probes, exchange of signals among the probes, and the linkage between the probe control and measuring system. Since the development of these techniques requires a vast amount of accumulated know-how and a very long period of time, it is difficult for a single measuring instrument manufacturer to develop them independently. The reality of the present situation is that the development of measuring instruments relies on the efforts of

Figure 3: Multi-probe System that enables the direct measurement of nano-structures

Source: Authors' compilation based on the chart provided by Dr. Tomonobu Nakayama, associate director of the National Institute for Materials Science

some researchers. In the following section of this report, we will introduce some application examples of the Multi-probe System and elucidate the role of the Multi-probe System in the fields of nanotechnology and biotechnology.

3 Application examples of the Multi-probe System

3.1 Direct measurement of electrical conductivity of nanomaterials

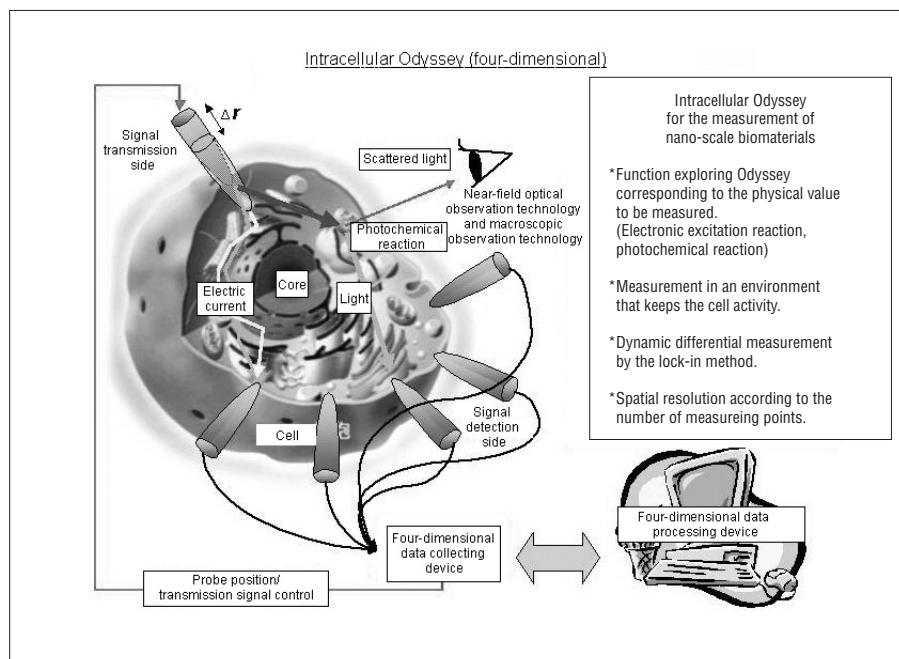
As a result of the recent progress in nanotechnology, research and development of nano-devices using nano-materials such as carbon nanotubes and DNAs has become a boom. In these studies, the measurement of the functions of nano-materials used is one of the most important issues. There are various kinds of carbon nanotubes, for example, and it has been reported that they show properties of metals, semiconductors, or insulators depending on their types. Therefore, it is very important to know the characteristics of the carbon nanotubes being used for a device. However, there is no established method to determine the characteristics. In an extreme case of the electrical conductivity of DNAs, there are various contradicting theories and it is not an exaggeration to say that nothing has been clarified.

To directly measure the electrical conductivity of these nano-materials, measuring systems

suitable for the size of materials are required (see Figure 3). The resistance of a resistor of a size of about 1 cm used for a general electrical appliance can be measured by a common circuit tester. To measure the characteristics of semiconductor devices, an instrument called a “prober” provided with probes having fine tips on the micron-scale is required. Furthermore, to measure the characteristics of nano-scale structures, the Multi-probe System provided with probes having fine tips on the nano-scale is required, which is now being developed by researchers.

The first measurement of the characteristics of nano-materials was carried out by a group from the National Institute for Materials Science, which was a measurement of electrical conductivity of a piece of fine ErSi₂ wire having a width of only 3 nm^[6]. ErSi₂, which was the material of the fine wire they used as the sample for measurement, exhibits the electrical conductivity characteristic of metals when it has large dimensions in a bulk state. Although the results of the measurement of the electrical conductivity of the fine wire showed a characteristic of metals, the values actually obtained were on an order of magnitude larger than those expected from the values in a bulk state. This is proof of the fact that electrical conductivity shows special characteristics in the nano-scale range. As shown in this example of electrical conductivity, characteristics of nano-materials are not on the extension of conventional materials, and the measurement of

Figure 4: Schematic diagram for the measurement of the functions of biomaterials using the Multi-probe System



Source: Prepared by Dr. Tomonobu Nakayama, associate director of the National Institute for Materials Science

characteristics is indispensable when using these materials. At present, the Multi-probe System is considered to be the only means that enables such measurement.

3.2 Direct measurement of the functions of biomaterials

The Multi-probe System is expected to contribute not only to the development of nano-devices that use biomaterials but also to the development of medical technologies. For example, it is expected that the Multi-probe System will enable the elucidation of the functions of biomaterials expressed in the antibody antigen response and the mechanism of the ingenious signal transmission and material transportation in biomaterials.

While the fine probes are brought to contact with the surface of materials in the above-mentioned measurement of electrical conductivity of nanomaterials, it becomes necessary in the measurement of the functions of biomaterials to obtain information of the inside of the material. In order to realize this, thin-long probes made of carbon nanotubes and metallic nano-rods are being investigated, as well as the means to extract the information obtained from the probe tips (inside of living organisms) by covering the portion other than the probe tips

with insulating material. Also being studied is the manner to detect specific antibody antigen responses from the change in mass caused by the response of attaching a particular biomaterial such as antibody to the probe tips. The use of a probe that detects light will enable the detection of chemical reactions by catching luminescence. By arranging such probes in a three-dimensional space, and measuring with the elapse of time, it will become possible to measure functions of biomaterials in four dimensions.

Figure 4 illustrates a schematic diagram of a Multi-probe System used for measuring functions of biomaterials. In this project called the "Intracellular Odyssey," signals are input into cells using a Multi-probe System and space-time distribution data (four-dimensional information) such as current are measured in order to catch the changes in cells corresponding to the input signals. It is expected that functions of biomaterials can be accurately measured by realizing the measurement in an environment that keeps the activity of cells.

3.3 Application to the development of existing semiconductor devices

In addition to the creation of new research fields in the nanotechnology and biotechnology areas, the Multi-probe System is expected to

contribute significantly to the development of existing semiconductor devices.

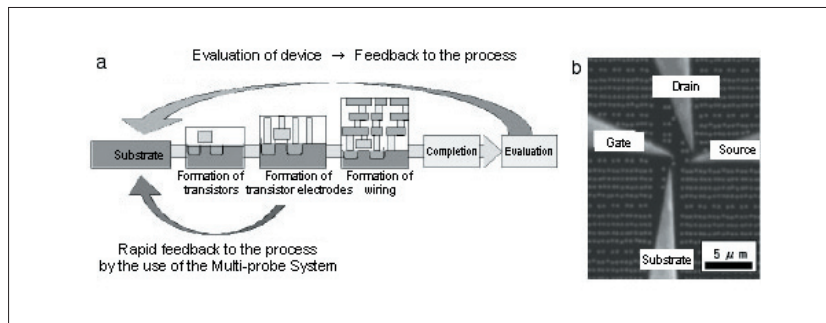
In the process of present semiconductor development, the loops of “design-manufacturing-assessment-change of design and manufacturing process” (the loop shown in the upper part of Figure 5) are repeated. In the past, the performance of a device had been evaluated by the finished product after the completion of the manufacturing process. This process from the start of production to the completion of assessment requires about 50 days. To reduce this long period required for the development of a device, Hitachi, Ltd. and Renesas Technology Corporation are trying to utilize the Multi-probe System for the assessment of performance during the manufacturing process before a product is completed^[7, 8]. The use of the Multi-probe System enables the confirmation of the performance of a device when manufacturing of the main structures including transistors have been completed, making it fast to generate

feedback for the changes in the design and manufacturing process (the loop shown in the lower part of Figure 5). By adopting this process, the assessment can be made within about 20 days after the start of manufacturing, so that the period required for the loop of a device’s development is reduced to half or less. This means that when the period required for development is half a year at present, it can be reduced to three months. Since the speed of product development and the improvement of yield (ratio of accepted products) are directly linked to profits, effects of the reduction of the period required for product development realized by the introduction of the Multi-probe System are significant.

4 Status of development in Japan and overseas

Table 1 shows major organizations that are carrying out the development of the Multi-probe

Figure 5:Reduction of the period required for the development cycle by use of the Multi-probe System



Source: Authors’ compilation based on the chart provided by Hitachi, Ltd., Renesas Technology Corporation, and Hitachi High-Technologies.
 a: Conceptual diagram for the reduction of the development cycle
 b: Probes brought to contact with a transistor electrode

Table 1:Major organizations that are carrying out the development of the Multi-probe System

	Research institute	Measuring instrument manufacturer
Domestic	National Institute for Materials Science* The University of Tokyo* Hitachi, Ltd.* Fuji Xerox Co., Ltd.* Osaka University* National Institute of Advanced Industrial Science and Technology Toyota Technological Institute Tokyo Institute of Technology	JEOL Ltd. Hitachi High-Technologies Unisoku Co., Ltd.
Foreign	University of North Carolina (U.S.A.) Harvard University (U.S.A.) University of Wisconsin (U.S.A.) Denmark Institute of Technology Seoul National University (Korea)	Omicron (Germany)

* Organizations that have disclosed actual data

System. Both in Japan and foreign countries, most of them are research institutes (including private research institutes). This fact indicates that researchers themselves are developing equipment in order to carry out the most-advanced research and development work. The Multi-probe System is often thought to be a simple device constructed only by adding a plural number of probes to the SPM. However, the difficulty of development is much greater because the subject of measurement has been changed from structures to functions. For this reason, some research institutes have given up on continuing research due to this difficulty. However, the number of institutes that challenge the difficulty, stimulated by the report mentioned in the preceding section, is definitely increasing.

Table 1 shows that the development is being carried out by many Japanese organizations. This comes from the fact that the first successful prototype was completed by a Japanese research institute. In particular, actual data have been disclosed only by Japanese organizations. That is, Japan is now leading the world in the development of equipment at the research level.

As for the status of development carried out by domestic and foreign manufacturers, however, domestic manufacturers are dominant in number, but Omicron (Germany), which is the only foreign manufacturer that is carrying out the development, has already completed a prototype and started sales. Although Japanese manufacturers are promoting the development cooperating with research institutes, they have not yet reached the level of commercialization.

In the Multi-probe System, it is important how to control a plural number of probes in the measuring procedure as well as the accumulation of tremendous know-how that can be obtained only by actual application of the device in research work. This means that both the ability to carry out research work and that to develop equipment are required. If a measuring instrument manufacturer constructs a device by simply mounting a plural number of probes, the device will not function as a Multi-probe System. In the development of a most-advanced device, manufacturers often provide the market (researchers) with prototypes

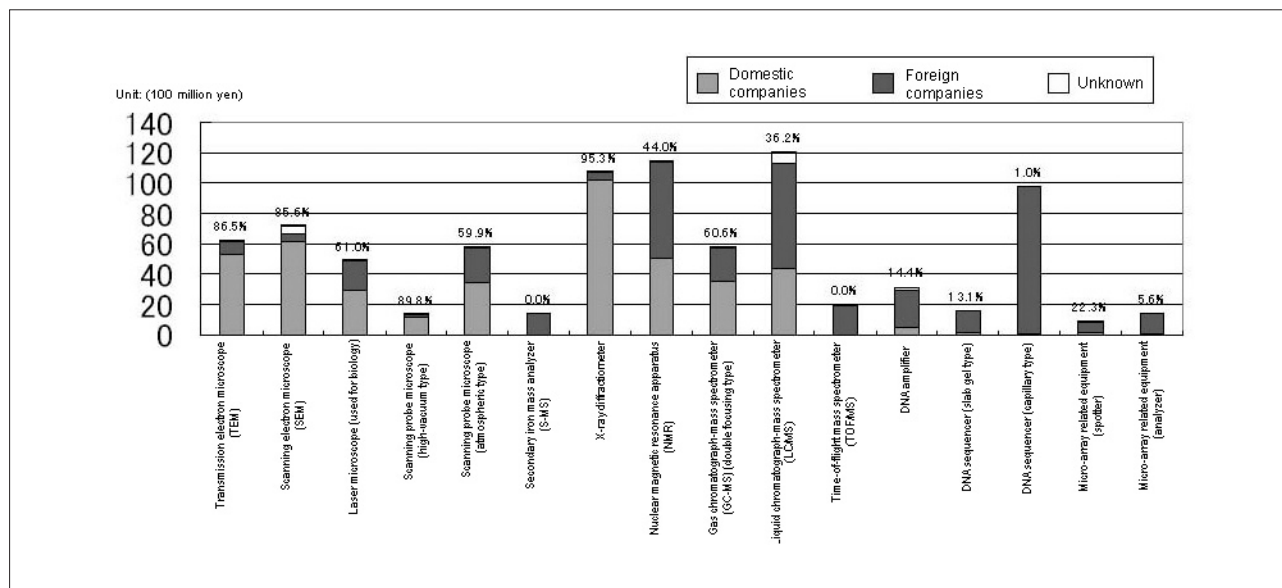
and obtain feedback from the market in order to complete the products with higher quality. This is certainly the case with the development of the Multi-probe System. The reason why no data have been reported from research institutes that have just purchased instruments available on the market is that the performance of instruments is not yet satisfactory. Table 1 does not include those research institutes that use purchased instruments available on the market.

The know-how indispensable for the development of the Multi-probe System has been sufficiently accumulated in Japan because research institutes are leading the world in the development, and, as such, Japan has a good chance to catch up and lead in the competition of commercialization. To realize this, it is necessary to promote the development with close cooperation between researchers and equipment developers.

5 Importance of the development of new measuring and analytical instruments

Most-advanced measuring and analytical instruments are indispensable for leading foreign countries in research and development and early commercialization of new technologies. In many cases, creative research results are supported by the most-advanced instruments that have been developed independently. The fact that many of the researches that have been awarded the Noble Prize have been integrated into the development of new measuring methods also indicates this. An immediate example is carbon nanotubes that were discovered in Japan and are considered to play a very important role in nanotechnology. This discovery was very much dependent on the electron microscope technology, which is one of Japan's specialties.

Unfortunately, however, many of the measuring and analytical instruments being used in the present workplaces for research in Japan are those that have been developed and commercialized in foreign countries. This is particularly true for the most-advanced equipment. Figure 6 shows the share of domestic and foreign companies for major most-advanced

Figure 6: Share of domestic and foreign manufacturers in major most-advanced measuring and analytical instruments (FY 2001)

Source: Prepared by the Research Environment and Industrial Relations Division, Research Promotion Bureau, Ministry of Education, Culture, Sports, Science and Technology based on the "Science and Technology Year Book" (published by R & D Corp., 2002)

instruments that are used in Japan. The chart shows that the most-advanced instruments used in biomaterials, which belong to one of the most important fields of research and development, are almost monopolized by foreign companies. Even with the instruments, the market share of which is occupied by domestic manufacturers, many of the high-performance instruments are provided by foreign manufacturers, and the scanning probe microscope, which is the basic technology of the Multi-probe System we are talking about, is an example of such cases.

It should be noted that procurement of most-advanced measuring and analytical equipment of foreign make leads not only to the outflow of budget but also to defeat in the fields of research and development. From this point of view, measures to promote the development of most-advanced measuring and analytical equipment are being investigated by the "Commission investigating the development of advanced analytical technology and instruments" (Ministry of Education, Culture, Sports, Science and Technology) and other organizations, and it is strongly desired that these measures are taken without fail as quickly as possible. We would like to point out that emphasis must be placed, under such development, on new devices

and instruments that may contribute to the exploitation of new research and development fields with originality as well as on those that provide the research and development fields with new methods by improving conventional devices and instruments.

Particularly in the efforts to improve conventional devices and instruments, it is important to judge whether the development can contribute towards opening a road to new research and development fields. In the history of the development of the electron microscope, for example, the improvement of resolution power from 1 nm to 0.1 nm gave much stronger impact to the fields of research and development than that from 10 nm to 1 nm, although both improvements are expressed by the same factor of 10. The former made it possible to observe atoms for the first time and opened the road to the technology that is called nanotechnology today. The above-mentioned STM had a prototype instrument called a "topografiner"^[9]. Although its constitution was almost the same as that of the STM, its resolving power was at the sub-micron level and it could not give such impact to the research fields that the STM did. The STM is now widely used because resolving power at the atomic level has been realized.

6 Conclusion

In order to lead foreign countries in the fields of research and development, it is indispensable to develop new, unique measuring and analytical equipment with originality. In the fields of nanotechnology and biotechnology, for example, completely new technologies and products different from conventional ones are being developed by making use of the unique functions of new materials such as biomaterials and nanomaterials. To achieve such objectives, the first and foremost thing to be done is to obtain knowledge of the functions that those new materials have, and the measurement of the functions cannot be done with conventional apparatus. In this report, we have introduced the Multi-probe System that is expected to pave the road toward creating new methods of research and, consequently, to create new research fields.

The Multi-probe System is a device in which the number of probes of the Scanning Probe Microscope (SPM) is increased. By the integrated control of a plural number of probes, direct measurement at the nano-scale level is enabled, which even the SPM could not achieve. Fortunately, Japan is leading the world in the development of this apparatus that has just started. The key to the success of the Multi-probe System lies in the development of the integrated control method of a plural number of probes including the development of measuring methods. In the development of the Multi-probe System, therefore, it is required for researchers, the users, and the device makers to closely cooperate more than in any other conventional devices and instruments. For Japan to overwhelm other nations in the fields of nanotechnology and biotechnology taking the advantage of

the present lead, it is strongly desired to take national measures that promote the research and development in these fields.

Acknowledgements

We would like to express our gratitude to Dr. Tomonobu Nakayama of the National Institute for Materials Science and Dr. Yasuhiro Mitsui of Hitachi High-Technologies for the relevant advice and precious materials that they have provided.

References

- [1] http://www.sk.icrr.u-tokyo.ac.jp/index_j.html
- [2] <http://www.shimadzu.co.jp/aboutus/nobel/>
- [3] Seizo Morita, "Scanning probe microscope," Maruzen. (in Japanese)
- [4] http://www.1.ibm.co.ibm/history/catalog/itemdetai_802000213.html
- [5] G. Binnig, H. Rohrer, Ch. Gerber and E. Weibel, "7×7 reconstruction on Si (111) resolved in real space," *Phys. Rev. Lett.* 50, 120 (1982).
- [6] Masakazu Aono et al., "Nano-wire Electrical Conductivity," *Japanese Scientific Monthly*, 54, 989 (2001). (in Japanese)
- [7] Y. Mitsui et al., "Physical and Chemical Analytical Instruments for Failure Analyses in G-bit Devices," *International Electron Devices Meeting 1998, Technical Digest, IEEE*, 1998, pp. 329-32.
- [8] Takahiro Onai et al., "Outlook for Advanced Semiconductor Process and Manufacturing," *The Hitachi Hyoron*, p. 5, April 2003. (in Japanese)
- [9] R. D. Young, J. Ward and F. Scire, "The topografiner: An instrument for measuring surface micro-topography," *Rev. Sci. Instrum.* 43, 999 (1972).