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〈기술해설〉

The Recent Activities in the XHV (Extreme High Vacuum) Techinque in Japan*

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1. Introduction

The definition of technical term of "vacuum" says that "vaccum is a pressure condition of lower than the atmospheric pressure". This means that "vaccum" has a pressure range as wide as more than the order of 10^{-15}. Usually, we classify this wide pressure range into five. These are, as shown in Table I, low vaccum, medium vaccum, high vaccum, ultra-high vaccum and extreme high vaccum. We can physically understand this classification as follows:

(1) As a pressure of 10^{-1} Pa, which is the boundary of medium vaccum and high vaccum, mean free path of gas molecule is about 10 cm. The length of 10 cm is almost the same order of ordinary vacuum devices, i.e., all phenomena are collision dominate in the condition of medium vacuum or low vacuum (the pressure range higher than 10^{-1} Pa), while in the high vacuum pressure region, all phenomena in the gas the almost collision free.

(2) At a pressure of 10^{-5} Pa, which is the boundary of high vaccum and ultra-high vaccum pressure regions, the molecular flux coming on the surface is

$$\Gamma = 3.0 \times 10^{18}\, p_0$$

for nitrogen gas under room temperature. Where p_0 is pressure in Pa. Remembering that the surface molecular density is about 10^{15}, (cm^{-2}) this means that, if the sticking probability of impinging molecules is 1, it will take about 30 sec to complete one monomolecular layer on the surface. It can be said that at the pressure of 10^{-5} Pa, the life time of clean surface is about 30 sec. This means that, if we need completely clean surface condition, it is necessary to prepare a vaccum lower than 10^{-5} Pa, i.e., ultrahigh vaccum is the least necessary condition to make investigations or some treatments on a clean surface.

(3) At a pressure of 10^{-9} Pa, which is the boundary of ultrahigh vacuum and extreme high vacuum (XHV), the impinging rate of gas molecules N on a small limited patch is given as,

$$N = 3 \times 10^{-6}\, n_0\, (s^{-1})$$

where n_0 is the number of molecules on this patch. This means, if we try micro-machining on a small surface of atomic size of about 100×100 atomic lattice for instance, the impinging rate of gas molecules is about 1 impinge per every 30 second. At a pressure of 10^{-9} Pa, we are free from impingement of gas molecule for about 30 sec to do something on the patch. The larger the working area is, the shorter the impinge free time to work with. Therefore, XHV technique is a necessary tool in promoting nanometer science and technology.

In Japan, a project to promote XHV technology has started in 1987 under the leadership of Science and Technology Agency of Japan. The final goal of this project is to establish fundamental techniques for the realization, measurement and application of XHV. The project has three groups, that is (1) XHV production group, (2) XHV measurement group and (3) Utilization of XHV group. A brief survey of activities of these groups is as follows.

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Table 1. Classification of Vacucum and its corresponding physical quantities

Clasification of vacuum	LV	MV	HV	UHV	XHV	
p [Pa]	10^5	10^2	10^{-1}	10^{-5}	10^{-9}	Pressure
λ [m]	10^{-7}	10^{-4}	0.1	10^3	10^7	Mean free path
Γ [s^{-1}]	3×10^{23}	3×10^{20}	3×10^{17}	3×10^{13}	3×10^9	Molecular flux per 1 cm^2
τ_s [s]	3×10^{-9}	3×10^{-6}	3×10^{-3}	30	3×10^5	average life of clean surface
$f_c\times10^4$ [s^{-1}]	3×10^{12}	3×10^9	3×10^6	3×10^2	3×10^{-2}	f_c: frequency of impingement of gas molecule as seen from a surface molecule
τ_c [s]	3×10^{-13}	3×10^{-10}	3×10^{-7}	3×10^{-3}	30	Mean free time from molecular impingement as seen from a small surface patch of $10^2\times10^2$ molelcular sites

2. XHV production group

The group has six working teams. The titles with short summaries are as follows.

(1) Improvement of sputter ion pump (SIP) for XHV use (S. Ukai *et al.*, Anelva Corporation).

By using a highly purified tantalum (99.99%) cathode, they have accomplished to get a pressure as low as 1.9×10^{-9} Pa without NEG and 1.7×10^{-9} Pa with a NEG. They are also trying to stabilize the PIG discharge in SIP under an XHV condition by adopting post-type cathode instead of plane cathode.

(2) Improvement of cryopump performances by lowering cryopanel temperature down to 3K level (H. Yamakawa *et al.*, ULVAC Japan Ltd.).

A cryopump of 10 inches in diameter with 3.5 K cryopanel has been completed. They certified that the final pressure is lower than 1.0×10^{-10} Pa after a baking of 400℃, 11 hours. They are now trying to measure the accurate pumping speed in XHV pressure range by means of conductance modulation method (cf. next). They are also trying to determine fundamental physical properties of 3.5 K surface such as sticking and adsorption probabilities, sojourn time of hydrogen and helium molecules.

(3) Precise evaluation of pumping speed and outgassing in vaccum pump at a very low operating pressure by means of conductance modulation method (T. Okano *et al.*, Institute of Industrial Science, University of Tokyo).

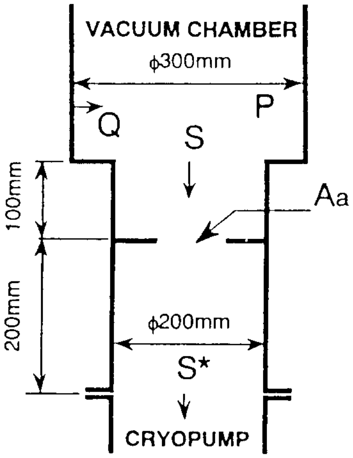

Fig. 1. A schematic diagram of conductance modulation method. By changing conductance of A_a, one can get true pumping speed S*.

The principle of the conductance modulation method is as follows. In a pumping system with a variable orifice between a test dome and a vacuum pump, they measure pressure p_c in the test dome as a function of inverse conductance 1/C of the variable orifice (see Fig. 1). If the gas desorption from the test dome is constant irrespective of the conductance of variable orifice, the relation between the pressure p_c and 1/C is linear, then the true pumping speed of the pump can easily be determined. By developing this method further, they have established an absolute calibration method of vacuum gauge in XHV pressure range, by introducting a known quantity of gas into the test dome by means of laser technique. This method can be applied to the performance evaluation of the above mentioned 3K level cryopump.

(4) Regulation and evaluation of outgassing rate from materials for XHV use (T. Homma, Chiba Institute of Technology).

A novel method of BN (boron nitride) coating has been developed to reduce outgassing rate from materials for vacuum use. By heating a special stainless steel which contains some amount of B and N, up to 870℃ for instance, B and N segregate on the surface and form a thin coating of BN. A test chamber of practical size of about 1.8 m length, 150 mm diameter will be prepared for the gas desorption rate measurement from the BN coated stainless steel.

(5) Dynamic regulation and evaluation of outgassing rate (T. Gotoh, Faculty of Science, Toho University).

Design and construction of outgas measuring system is now going on. The system will be equipped with a time of flight type partial pressure gauge and vacuum micro balance.

(6) Development of pure aluminum clad plate of aluminum alloy for XHV use (M. Tetsu *et al.*, Sukegawa Electric Co. Ltd.).

They are planning to get a clad plate of 99.99% pure aluminum and aluminum alloy. How to weld clad plate to form a vacuum tight chamber and how to join vacuum components by flanges are the main problems of this theme.

3. XHV measurement group

(1) Development of cold emitter as an electron source of XHV ionization gauge (Y. Ishizawa, National Institute for Research in Inorganic Materials).

It was certified that NbC (niobium carbide) has a good performance as an electron emitter. In addition, mono-molecular layer of graphite on NbC will improve the performance further. It was certified that an ethylene gas exposure of 300*l* at 1100℃ forms a good monomolecular layer of graphite on NbC (111) surface or ZrC (111) surface, and similarly, an exposure of 50000 L at 1250℃ also forms a good monomolecular graphite layer on NbC (100) surface. Now they are searching the best condition to get a good and stable cold emitter for an XHV ionization gauge.

(2) Development of a novel cold cathode gauge with very high sensitivity (T. Kanaji, Faculty of Science, Kobe University).

By applying a serial electrostatic lens system, a cold cathode gauge with very high sensitivity has been developed. A computer simulation traces a motion of electron in the gauge with an arbitrary initial condition, evaluates average totoal path length of electron emitted from cold cathode, and finally estimates the sensitivity of the gauge. At present, a sensitivity of about 15 (Pa^{-1}) has been accomplished in a preliminary test. A space charge effect of electrons causes some discrepancy between the computer simulation and the experiment. A more accurate simulation will be done taking into account the space charge effect.

(3) Development of a novel vaccum gauge with very low residual current limit (T. Oshima, School of Science and Engineering, Waseda University).

To eliminate all kinds of residual current, they introduced an idea of electrostatic deflection. They paid much efforts to decrease electron stimulated desorption and the well known soft X-ray current. They have introduced gold grid to form deflector electrode system, because the gold is the best material with very low ion desorption rate. Grid structure itself also reduces ion desorption rate (Fig. 2). At present, pressure measurement has been successfully done down to 1.7×10^{-11} Pa with a channeltron as the ion current detector.

(4) Development of a novel vacuum gauge by means of laser excitation method (K. Kokubun *et al.*, Electrotechnical Laboratory).

The principle of the gauge is to ionize gas molecules by a focused intense laser beam. Because the photon energy of the laser is not enough to ionize gas molecule, we expect multi-photon ionization process in the intense laser light. The laser used is ArF excimer-laser. One of the problems is scattering of laser light. Some of the scattered laser light hits a dinode electrode of electron multiplier and emits secondary electrons which cause a noise. Because of a limited power and size of laser beam,

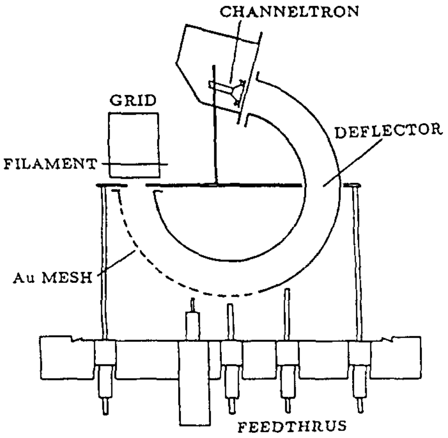

Fig. 2. A schematic diagram of a novel ionization gauge of electrostatic deflection type with very low residual current.

the size of active space is very small, where the photon density of laser light is high enough to ionize gas molecules by multi-photon ionization process, and as a result, the output ion current is very small. For the measurement of small ion current, they have developed and successfully completed an ion counting method by means of micro channel plate and CCD. By this method, we can count the number of ions produced in the active space down to several tens per laser shot. Now, we have certified a linear relation between pressure and the output current of the gauge down to 1×10^{-8} Pa.

4. XHV utilization group

(1) Development and evaluation of micro-actuator (H. Fujita *et al.*, Institute of Industrial Science, University of Tokyo).

As a fundamental mechanism of micro-machine of nanometer size, they have developed a fundamental component to give a small displacement by applying micro-machining technique which has been developed in the VLSI industry. The device is composed of a electrostatic actuator, an elastic suspension mechanisms and a tunneling current detector as a displacement sensor.

(2) Development of lubricative surface in XHV (K. Yoshihara *et al.*, National Research Institute for Metals).

By means of segregation of boron and nitrogen onto surface, formation of BN thin layer was tested. A BN thin layer largely reduces frictional froces under an XHV condition.

(3) Production and evaluation of ultra-clean surface (K. Odaka, Hitachi Ltd.).

A testing system has been constructed. In the system, an electron gun of 30 mA in current and of 1-3 kV in energy, a surface analyzer of electron diffraction type and a three-fold air lock system are equipped. Using the air-lock system, one can mount the test piece in the XHV chamber in 2 hours without breaking the vaccum.

Although the governmental support for this activity will end by this March, the activity to develop XHV technique will be kept. In the spring of 1994, a small work shop on XHV technique will be held in Japan, where the all above mentioned activities will be summarized and be opened to be discussed. We are expecting that these activities will become one of the main topics in the field of nanometer science and technique at the International Congress IVC-13/ICSS-9 in Yokohama. IUVSTA has, as you know, decided to organize nanometer science and technique division as the 8th division of STD from the next triennium.