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Pumping experiment of water on B and LaB_6 films with electron beam evaporator

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Abstract

Pumping characteristics of water vapor on boron and lanthanum hexaboride films formed with an electron beam evaporator have been investigated in high vacuum of a pressure region between 10^{-4} and 10^{-3} Pa. Measured initial maximum pumping speeds of water for fresh B and LaB_6 films on substrates with a deposition amount from 2.3×10^{21} to 6.7×10^{21} molecules$\cdot m^{-2}$ are 3.2 ~ 4.9 $m^3\cdot s^{-1}\cdot m^{-2}$, and maximum saturation amounts of adsorbed water on these films are 2.9×10^{20} ~ 1.3×10^{21} H_2O molecules$\cdot m^{-2}$.

Key words: LHD,boronization, boron, lanthanum hexaboride, getter action for water, pumping speed of water, electron beam evaporation

1. Introduction

From the objective of oxygen impurity control in fusion devices boronization has been recently developed as a useful wall conditioning technique[1] and has been successfully applied in a large machine, such as DIII-D[2],[3]. A super conductive large helical device (LHD) is now under construction[4] at National Institute for Fusion Science (NIFS). The allowable baking temperature of the vacuum chamber of this device is limited to around 100°C from a technical requirement to keep structural deformation of the vacuum chamber due to thermal expansion as low as possible. For the low temperature baking, as a matter of course, it is very important to reduce effectively adsorbed water on the chamber wall with any wall conditioning technique, because the water becomes a main source of oxygen impurity. A noticeable fact that adsorbed water on the wall of non-bakable port hall in TEXTOR is well removed by only introduction of diborane gas into the vessel has been suggested by Waelbroeck[5]. We have focused on this point. The objective of this study is to investigate in detail the water pumping action of boron film, and to get scope for achievement of water free ultrahigh vacuum in LHD, because the application of boronization is at present taken into account as a candidate technique for the wall conditioning of LHD and available data on water pumping of boron film are few. In the first study a solid sample of lanthanum hexaboride (LaB_6) has been physically evaporated in a vacuum chamber instead of use of diborane gas, and the pumping action for residual water on the deposited surface has been observed. As a result of pumping action for residual water on the deposited wall, improvement of base pressure of the chamber has been observed[6]. The LaB_6 sample is simply selected because of easy evaporation

compared with a solid boron[7]. In this paper recent results of quantitative measurement of pumping speeds of water on a boron or lanthanum hexaboride deposited surface are described.

2. Experimental

2.1 Experimental apparatus

A vacuum coater is used for water pumping experiment. The schematic illustration of the coater is shown in Figure 1. The chamber of coater is made of 304 stainless steel and is 560 mm in diam. and 385 mm in length. The pumping system consists of a compound molecular pump(Type TG-200, Osaka vacuum Ltd.,) of nominal pumping speed of 0.2 $m^3 \cdot s^{-1}$ for nitrogen, and a mechanical pump. Total and partial pressures of the pumped chamber are monitored with a conventional ionization gauge and a gas analyzer(QMG064, BALZERS), respectively. Distilled water is introduced to the chamber through a variable leak valve on the measurement of water pumping speed. A 270° deflection type electron beam evaporator(model JEBG-203UB, JEOL Co.,) and a couple of substrates are set in the chamber. The one of substrates is a short glass tube of 160 mm in diam. and 220 mm in length, and the other one is a copper plate set at the upper end of the glass substrate. The effective substrate area of deposition viewed from the evaporation source, As, is As=0.13 m^2. Evaporation samples which are commercially available are a lump of boron(99.9 % purity) and a circular disk plate of lanthanum hexaboride(30 mm in diam. and 3.4 mm in thickness, 99.9 % purity).

2.2 Pumping experiment of water

After achieving of a base pressure of 2.7×10^{-4} Pa in the coater, water is introduced to the chamber at a constant pressure, P_1 between 2.7×10^{-4} Pa and 2.7×10^{-3} Pa and then the sample of B or LaB_6 is evaporated on the substrate for 2 min at the heating power of 560 W (7kV×80mA). The temperature of copper substrate after evaporation of each sample is monitored with a thermocouple, and the typical temperature is about 60 °C. Figure 2 (a) and (b) shows time evolutions of typical pumping down curve of total pressure, P(t) and ion current of water, $I^+(H_2O)$ observed with the total pressure gauge and the gas analyzer during evaporation experiments of B and LaB_6 samples. Since release of hydrogen gas is mainly observed during the evaporation, change of output ion current of hydrogen, $I^+(H_2)$ is shown in Fig.2 together with that of water, $I^+(H_2O)$.

The pumping speed of water generated on the substrate surface as a result of the B or LaB_6 evaporation can be estimated with following pumping equations:

before evaporation $$Q(W)+Q(H_2O)=S_pP_1 \quad (1)$$

after evaporation $$Q(W)+Q(H_2O)=(S_p+S_g)P(t) \quad (2)$$

where $Q(W)$ and $Q(H_2O)$ are the total outgassing rate from the chamber wall and the constant throughput of water, respectively, and S_p is the effective pumping speed(N_2 equivalent) of the compound molecular pump at the pumping port of the chamber taking the value of $S_p=0.17\ m^3\cdot s^{-1}$, S_g is the pumping speed(N_2 equivalent) of water generated at the substrate (the getter surface) due to the coating of B and LaB_6. From Eq.(1) and (2), the pumping speed, S_g is given as

$$S_g=(P_1/P(t)-1)S_p. \quad (3)$$

In this experiment total pressure ratio between P_1 and P(t) in Eq.(3) is replaced with output ion currents of water, $I_1^+(H_2O)$ and $I_t^+(H_2O)$ as $P_1/P(t)=I_1^+(H_2O)/I_t^+(H_2O)$. The absolute pumping speed for water, S is determined with multiplying S_g value of Eq.(3) by a root of the mass ratio between water and nitrogen as

$$S=\sqrt{m(N_2)/m(H_2O)}\cdot S_g. \quad (4)$$

In Eq.(3) the Sg value becomes maximum when the total pressure, P(t) takes a minimum. Here we define the maximum pumping speed after the evaporation of B or LaB_6 as the initial maximum pumping speed.

Figure 3 (a) and (b) are plots of initial maximum pumping speed of water, measured for the fresh gettered surfaces of B and LaB_6 under different water throughputs, where pumping speed of the vertical axis is normalized with the effective surface area as S/As. The pumping action of the gettered surface for water is kept until water adsorption on the surface reaches to saturate, and the pumping speed for water gradually decreases in accordance with the adsorption process. Figure 4 shows typical time variations of pumping speeds of water for the both getter surfaces of B and LaB_6 after the evaporation.

3. Discussion

Duration of water pumping of both films of B and LaB_6 can be observed from results of Fig.2 and Fig.4. The LaB_6 film keeps clearly longer pumping time by about 6 times than the B film. This difference may be caused by the difference between

evaporation amounts of both samples of B and LaB_6. In fact, as mentioned later, the evaporation amount of the LaB_6 sample is about 6 times as much as that of the B sample. The rapid decrease of the pumping speed of water after stop of the B evaporation in Fig.2 (a) or Fig.4 shows that the B gettered surface is nearly saturated with adsorbed water during the evaporation because of the small evaporation amount. Thus the duration of water pumping is depending on the water pumping capacity of gettered film which is proportional to the total evaporation amount of the sample per one time. Prior to every measurement of the pumping speed of water, samples are well degassed by preheating (7kV×30mA, 30min). Nevertheless, as seen in Fig.2, appreciable hydrogen gas release occurs during evaporation of both samples of B and LaB_6. It is presumed that the pumping capacity of B or LaB_6 film for water is reduced due to co-adsorption of the evolved hydrogen on the film. However, it is difficult at present to discuss the influence of the hydrogen co-adsorption to the water pumping in this experiment.

It is seen from results of Fig.3(a) and (b) that initial maximum pumping speeds of water for both fresh getter surfaces take nearly equal values. This fact shows that in the comparison of getter action between B and LaB6, the presence of lanthanum containing in the LaB_6 compound has no special contribution to water pumping. Our measured values of pumping speeds of water on B and LaB6 getter films are comparable with values (around 3 $m^3 \cdot s^{-1} \cdot m^{-2}$) of pumping speed of hydrogen and deuterium on Ti-getter films at room temperature reported by Elsworth et al,[8]. In Fig.3 there are observed large scatterings between measured data of the pumping speed. The scattering may be caused from non-uniformity between deposition amounts in every evaporation. The

evaporation amount of each sample cannot be kept constant because of high melting point of the sample, although the heating power of electron beam is always kept constant every evaporation. The evaporation amount can be roughly estimated from weight loss of the sample after several runs of evaporation. The average evaporation amount per one time is 16 mg/run for the B sample and 100 mg/run for the LaB_6 sample, respectively.

The total adsorption amount of water on each gettered film during and after evaporation can be estimated from the time integration of pumping down curve as $\sqrt{m(N_2)/m(H_2O)}\cdot S_p\cdot\int(P_1-P(t))dt$. The typical calculated results are summarized in Table-1. In this table the film thickness is estimated from the weight loss of the sample and bulk density for B or LaB_6 ($\rho(B)=2.34 g\cdot cm^{-3}$, $\rho(LaB_6)=3.5 g\cdot cm^{-3}$, respectively). From the table average evaporation amount for the B sample is rather larger by a fact of 3 than that for the LaB_6 sample. But in the comparison of maximum total adsorption amount of water the B film takes smaller value by a factor of 4 than the LaB_6 film.

From the observation of strong pumping action of B and LaB_6 films for water in this experiment, we can assert that boron coating is a very promising technique for control of water impurity in fusion devices. And B or LaB_6 is a preferable material for a strong getter of water for general vacuum use.

4. Conclusion

The getter actions of evaporation films of boron and lanthanum hexaboride for water have been investigated at water pressures between 10^{-4} and 10^{-3} Pa at room

temperature. For the fresh films of evaporation amount of the order of 10^{21} B or LaB_6 atoms/m^2, typical values from 3 to 4 $m^3 \cdot s^{-1} \cdot m^{-2}$ are measured at the substrate temperature of 60 °C as the initial maximum pumping speed. The typical total adsorbed amounts of water on these films are the order of $10^{20} \sim 10^{21}$ H_2O molecules$\cdot m^{-2}$. The vacuum use of boron or lanthanum hexaboride material is an interesting subject in the future.

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Figure and table caption.

Figure.1: Schematic drawing of experimental apparatus.

Figure.2: Time evolution of total pressure and output ion currents of water and hydrogen for Boron evaporation (a) and LaB_6 evaporation (b).

Figure.3: Initial maximum pumping speed of water after B evaporation (a) and LaB_6 evaporation (b).

Figure.4: Time variations of pumping speeds of water on B and LaB_6 films.

Table-I: Results of water adsorption on B and LaB_6 films.

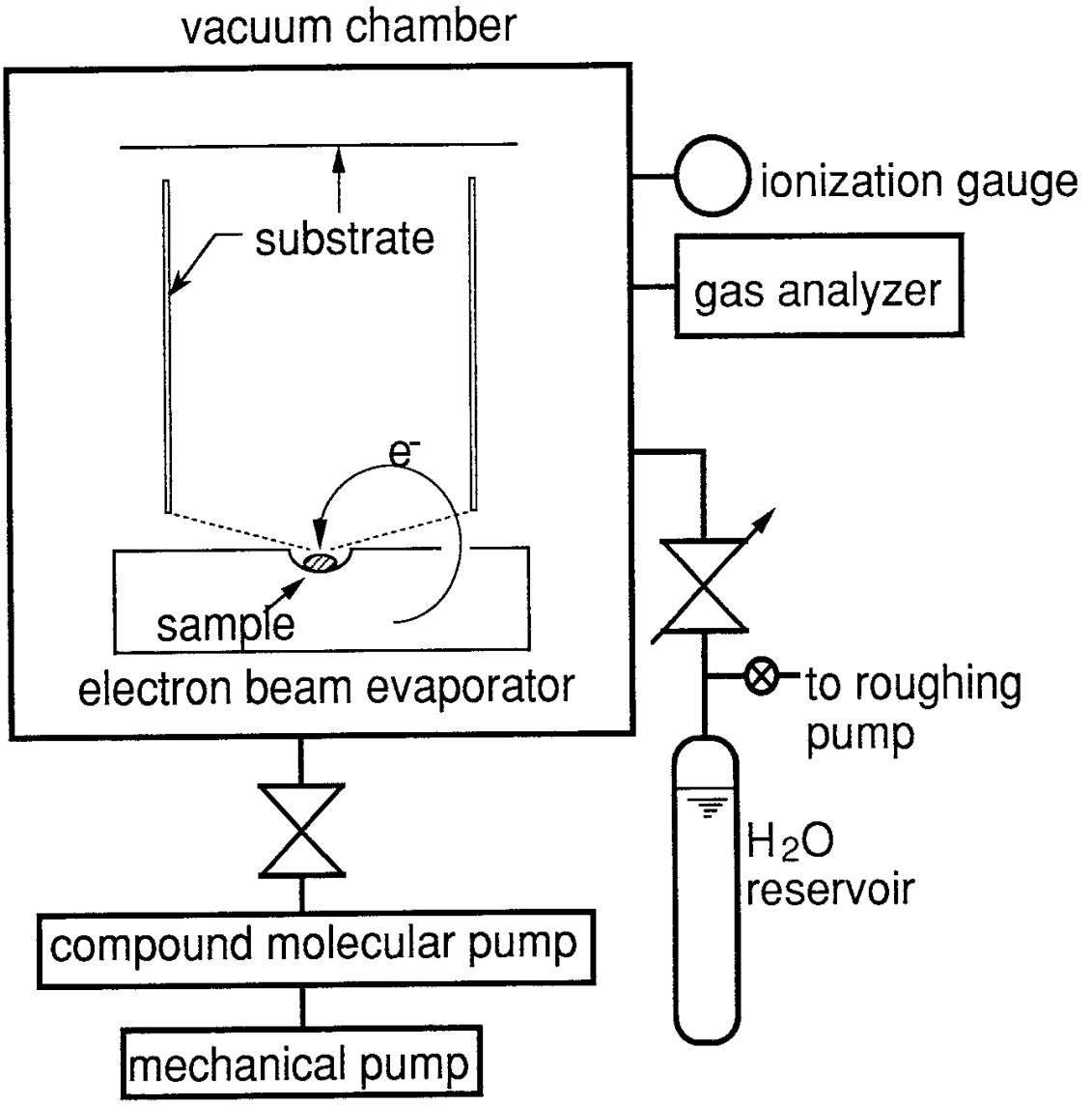
vacuum chamber
substrate
e⁻
sample
electron beam evaporator
ionization gauge
gas analyzer
to roughing pump
H2O reservoir
compound molecular pump
mechanical pump

Fig.1

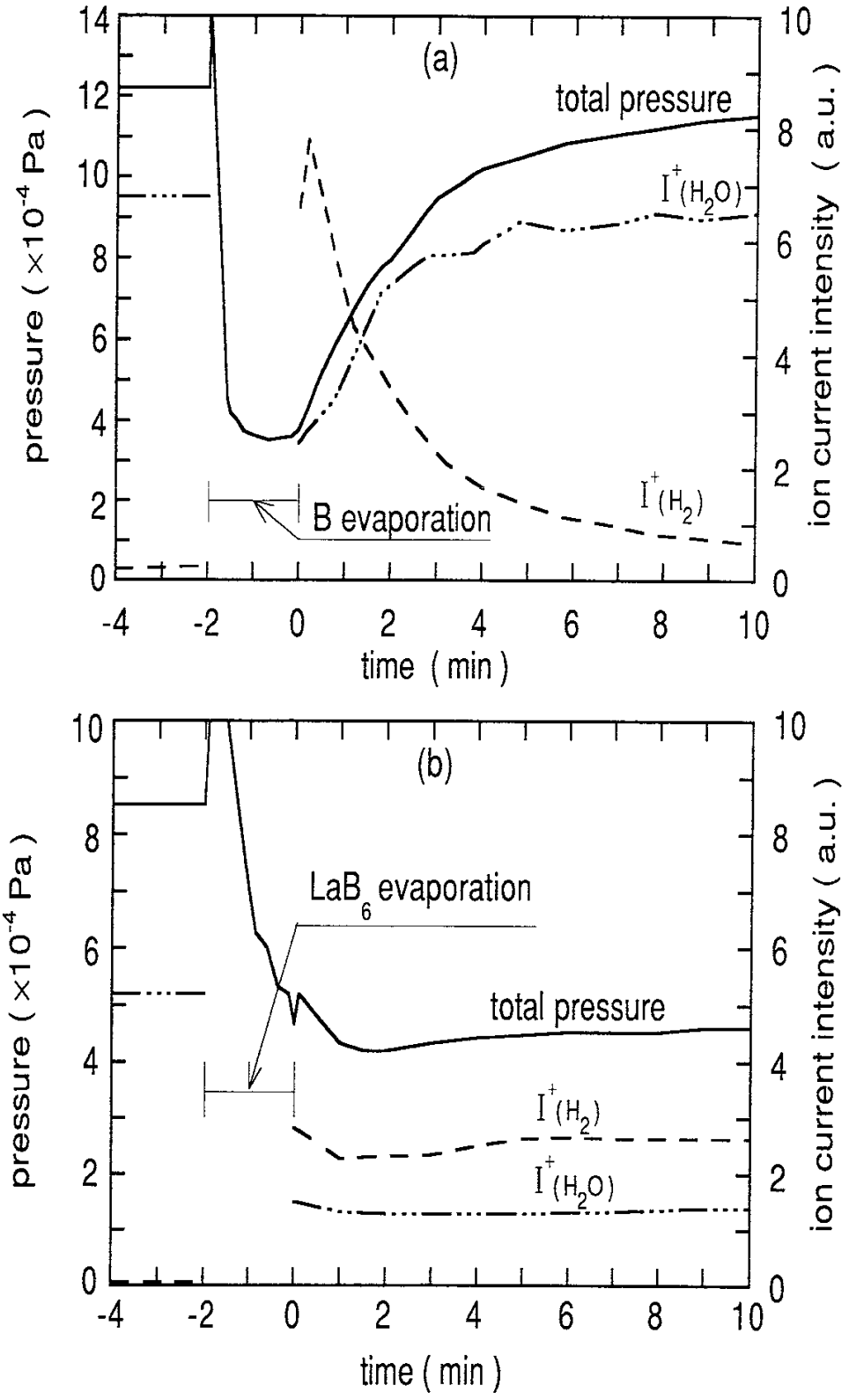
(a)
total pressure
$I^+(H_2O)$
$I^+(H_2)$
B evaporation
pressure ($\times 10^{-4}$ Pa)
ion current intensity (a.u.)
time (min)
(b)
LaB_6 evaporation
total pressure
$I^+(H_2)$
$I^+(H_2O)$
pressure ($\times 10^{-4}$ Pa)
ion current intensity (a.u.)
time (min)

Fig.2

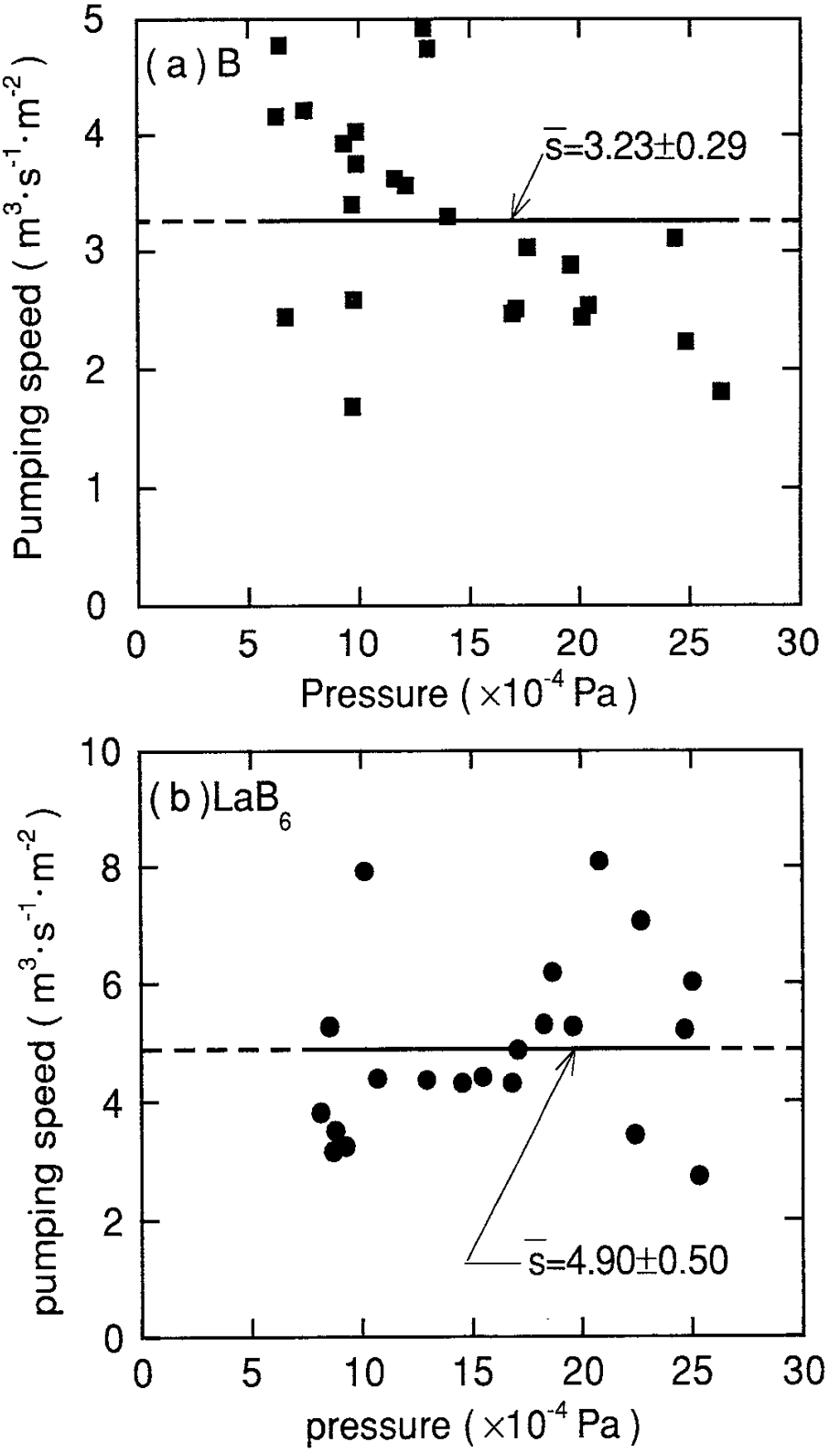
(a) B
$\bar{s}$=3.23±0.29
Pumping speed ($m^3 \cdot s^{-1} \cdot m^{-2}$)
Pressure ($\times 10^{-4}$ Pa)
0
1
2
3
4
5
0
5
10
15
20
25
30
(b) LaB_6
$\bar{s}$=4.90±0.50
pumping speed ($m^3 \cdot s^{-1} \cdot m^{-2}$)
pressure ($\times 10^{-4}$ Pa)
0
2
4
6
8
10
0
5
10
15
20
25
30

Fig.3

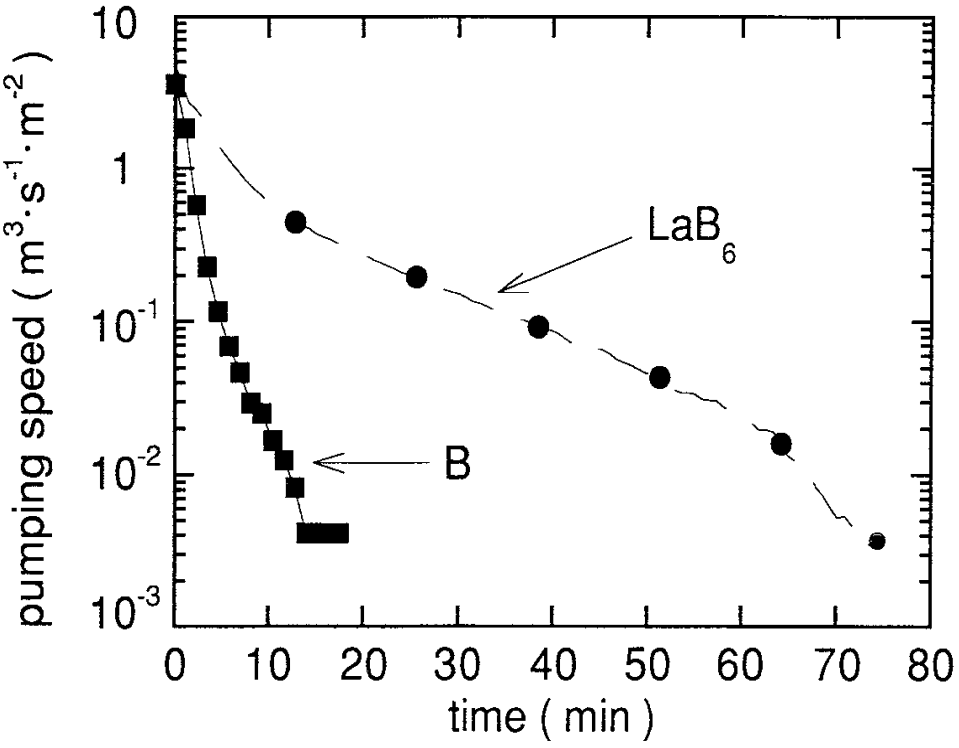
10
1
10^{-1}
10^{-2}
10^{-3}
pumping speed ($m^3 \cdot s^{-1} \cdot m^{-2}$)
0
10
20
30
40
50
60
70
80
time (min)
LaB_6
B

Fig.4

Table-I. Results of water adsorption on B and LaB_6 films.

film	average number of deposited molecules for 2 min evaporation ($\times 10^{21}$ molecules·m^{-2})	film thickness (nm)	total amount of adsorbed water on film ($\times 10^{21}$ molecules·m^{-2})	
			min.	max.
B	6.7	53	0.21	0.29
LaB_6	2.3	220	0.29	1.30

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