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# **Control of Discharge Conditions to Reduce Hydrogen Content in Low Z films Produced with DC Glow**

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## **ABSTRACT**

Boronization at near room temperature has been performed in plasma processing teststand (PPT) by using a 5 % diborane gases  $B_2H_6$  in He on electrically floating or unfloating Al samples under various conditions on DC glow discharge power or total gas pressure. The hydrogen concentration was analyzed by using elastic recoil detection method (ERD) and a new modified normalizing technique with Rutherford back scattering (RBS). Results showed that a high growth rate of film formation and floating surface were effective in reducing hydrogen concentration in B films. This result was in good agreement with earlier measurements of H with flash filament (FF) desorption method. In particular the H/B ratio was reduced by decreasing ions but increasing radicals for B film formation.

Key words: DC glow, boronization, elastic recoil detection, hydrogen content,  
H recycling, LHD.

## 1. INTRODUCTION

Control of hydrogen particle recycling on plasma facing materials is considered to be one of key issues to improve plasma performance in Large Helical Device [1,2]. In LHD, on the other hand, low Z coating such as boronization of wall surfaces is considered as the main technique of wall conditioning, because the vacuum vessel is allowed to be baked at only the temperature below 100 °C [3,4]. In this low temperature range, however, hydrogen atoms contained in low Z films formed with such as boronization or carbonization are known to be thermally desorbed not so sufficiently [5] as performed in TEXTOR [6], DIII-D [7], and JT-60U [8] at elevated wall temperature above 200 °C, resulting in good control of hydrogen recycling and significant improvement of the plasma parameters.

So far, in our experiments, the hydrogen content in boronized or carbonized films produced at room temperature was successfully reduced by controlling glow discharge conditions. The low Z coated film was produced using the gas mixtures of decaborane B<sub>10</sub>H<sub>14</sub> and He or pure CH<sub>4</sub> in DC glow discharge. The results showed that the hydrogen content decreased with the increase of the growth rate of film formation [9-10].

In our previous works the H content were measured by flash filament (FF) desorption method or residual gas analysis (RGA). FF was set at electrically floating potential to avoid heating up due to discharge current. In this work to make clear the difference between floating and unfloating, B coated films were prepared on floating or unfloating samples, and depth profiles of H were analyzed with ERD technique. For this purpose, new modified method using RBS was established to monitor the probing ion flux. The results using ERD were compared with conventional method of FF or RGA.

## 2. EXPERIMENTAL

### 2.1. Discharge conditions and film depositions

Experiments on controlling the hydrogen content were systematically performed by using Plasma Processing Teststand (PPT) facility. A schematic diagram of the experimental set-up is shown in Fig.1. The principle of the controlling discharge conditions and the experimental set-up have been described in elsewhere [9-10]. For the present work, two pure aluminum sample plates (99.999%) with the size of 20x10x1mm were set on the sample holder and placed on the cathode (liner) so that the sample surfaces to be exposed to a plasma were under the same geometrical condition. The surface temperatures of floating, unfloating, the liner, the quartz oscillator, the flash filament and anode were monitored with a thermocouple and an infrared TV camera in order to maintain liner and substrate surface temperature  $T$  below 150 °C, under which thermal desorption of H from B film is negligible [5]. Here, it is noted that, except for the quartz oscillator, all discharge systems were isolated from the ground to make the discharge at only the inside of the cathode.

After evacuated below  $10^{-6}$  Torr, sample gases  $B_2H_6$  and He were introduced into the chamber with typical flow rates in the range of 5 to 50 sccm and the pressure in the range of 60 to 200 mTorr by adjusting pumping speed. Coated films were prepared under DC glow discharge with the input power of 80 to 300 W at the voltage between 300 to 600 V or 0.2 A to 0.5 A. During the discharge the operation parameters were fixed at almost constant conditions, and the plasma was analyzed using a Langmuir probe before fully contaminated the probe surface under coating.

By considering the maximum sampling depth and depth resolution of ERD, the optimum thickness of boron film was estimated to be about 100 nm. During film formation for 5 ~ 15 min, however, the surface temperature of samples increased and discharge were stopped at temperature near 150 °C and cooled down. This procedure was repeated for 0 to few times to produce the 100 nm thick film depending on discharge conditions.

After each boronization, the system pumping speed was readjusted to 4 l/s with He, then the FF was flashed with monitoring desorbed H<sub>2</sub> with RGA. Lastly, the coated samples were taken out to atmosphere from the chamber for H content and thickness measurements with ion beam analysis as described below.

## 2. 2. Determination of hydrogen during film growth.

In order to measure hydrogen content and depth profiling the ion beam analysis of ERD by using 1.5 MeV He<sup>+</sup> ions was performed. Experimental set-up was the same with that described in [2,11]. The ion beam for depth profiling was 1.5 MeV He<sup>+</sup> impinging on the target making an the incident angle of the ion beam was set at 10 ° from the sample surface. A solid state detector of ERD for recoiled H atoms was placed at the forward scattering angle of 20 ° from the probe beam, with a mylar filter of 6 μm thick at the front of the detector to cut the scattered He ions from the target [11]. In order to measure the probing ion fluence, the RBS detector was set at 150 ° from the probe beam as shown in Fig.2.

## 3. RESULTS and DISCUSSION

Figure 2 shows a typical RBS spectrum taken from the unfloating B coated Al sample, on which B can't be clearly observed, probably due to

surface roughness of Al target. The B film thickness was measured from the shift of the Al surface edge as shown in Fig. 2. Then Al edge intensity with calculated scattering cross section  $\sigma$  and energy stopping power  $\epsilon$  under the B film was used as the measure of the probing He<sup>+</sup> fluences which was used to obtain absolute H concentration from ERD spectra. This method was newly established in this work for the use in the case of low Z thin films.

Figure 3 shows the hydrogen atom profiles in boron films formed on floating or unfloating Al samples. The ERD data processing program was calibrated by using a vanadium hydride (V<sub>2</sub>H) standard sample. The atomic density  $\rho_B$  of the B film was supposed to be 1.5 g/cm<sup>3</sup> [6]. From this figure it is observed that the H profile on the unfloated sample is more dense, flat, and thick in comparison with the floated one. These results will be discussed later.

Figures 4(a) and (b) shows the peak H/B atomic ratio and the film growth rate measured by ERD and RBS as a function of the discharge power and total gas pressure, respectively. From both figures it can be observed that the H/B are reduced with increasing the film growth rate when the discharge power is increased, or when the total pressure is increased. These results are in good agreement with our results obtained so far from the FF method or RGA [9-10].

The film growth rate gives the B atoms flux  $\phi_{BU}$  and  $\phi_{BF}$  for unfloating and floating cases, respectively. Here it may be supposed that  $\phi_{BU}$  consists of B<sub>n</sub>H<sub>m</sub> ions and neutrals, that is, radicals, and  $\phi_{BF}$  consists of mainly radicals, because, under glow discharge, ions are mainly produced in the cathode sheath due to electron impact and they are mostly extracted to the cathode surface which has deeper negative potential of about 500 V on the unfloating sample than that of a several V on the floating sample. Therefore  $\phi_{BU} - \phi_{BF}$  may give B atom flux in forms of B<sub>n</sub>H<sub>m</sub><sup>+</sup>. From Fig. 5 (a) and (b) it is clearly observed that the H/B ratio is reduced with reducing the ionic flux ( $\phi_{BU} - \phi_{BF}$ ). It is also

observed that, though the ionic flux contributes to increasing the film growth rate as shown in Fig. 4, it is better to increase the radical flux  $\phi_{BF}$  to reduce the H/B ratio.

To explain the difference between ions and radicals regarding growth of B film and reduction of H/B ratio, there must be considered two aspects, that is, the difference of molecular forms concerning B and H, and the difference of surface reactions such as impact desorption, recoil implantation and/or surface recombination of H atoms depending on kinetic energies of impinging particles including  $\text{He}^+$  ions. Within this work it is not easy to evaluate these aspects quantitatively, and more detailed experiments will be needed, comparing with complementary modeling calculations.

#### 4. CONCLUSION

Boronization at near room temperature was performed from diborane gases  $\text{B}_2\text{H}_6$  on electrically floating or unfloating Al samples under various conditions on DC glow discharge power or total gas pressure. The hydrogen concentration was analyzed by using elastic recoil detection method (ERD) and a new modified normalizing technique with Rutherford back scattering (RBS) was introduced. Results showed that a high growth rate of film formation and floating surface were effective in reducing hydrogen concentration in B films. This result was in good agreement with earlier measurements of H with flash filament (FF) desorption method. In particular, due to advantage of using floating and unfloating samples, contributions of ionic and radical fluxes were separately characterized, resulting that the H/B ratio is reduced by decreasing the ionic flux but increasing the radical one for B film formation.

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## FIGURE CAPTIONS :

Fig. 1. The schematic illustration of the PPT device with showing locations of the samples.

Fig. 2. RBS spectra taken from B films prepared on electrically unfloating Al samples.

Fig. 3. Depth profiles of hydrogen atom in boron films on electrically floating or unfloating Al samples.

Fig. 4(a). Discharge power dependence of H/B peak ratio in depth profile and growth rate on the floating or unfloating Al samples.

Fig. 4(b). Total gas pressure dependence of H/B peak ratio in depth profile and growth rate on the floating or unfloating Al samples.

Fig. 5(a). Relation between the H/B ratio and the B molecules flux on the floating or unfloating Al samples under the discharge power dependence.

Fig. 5(b). Relation between the H/B ratio and the B molecules flux on the floating or unfloating Al samples under the total discharge pressure dependence.

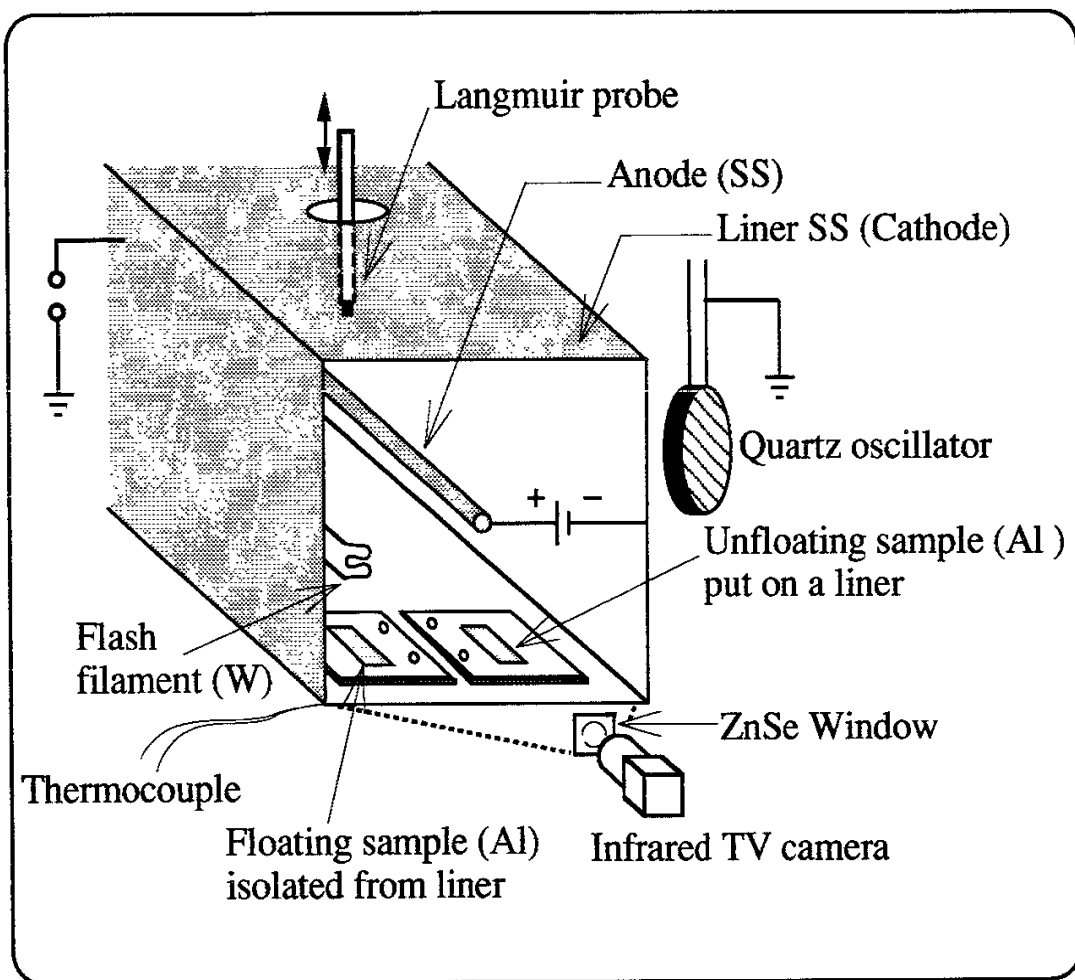


Figure 1, M. Natsir, et. al.

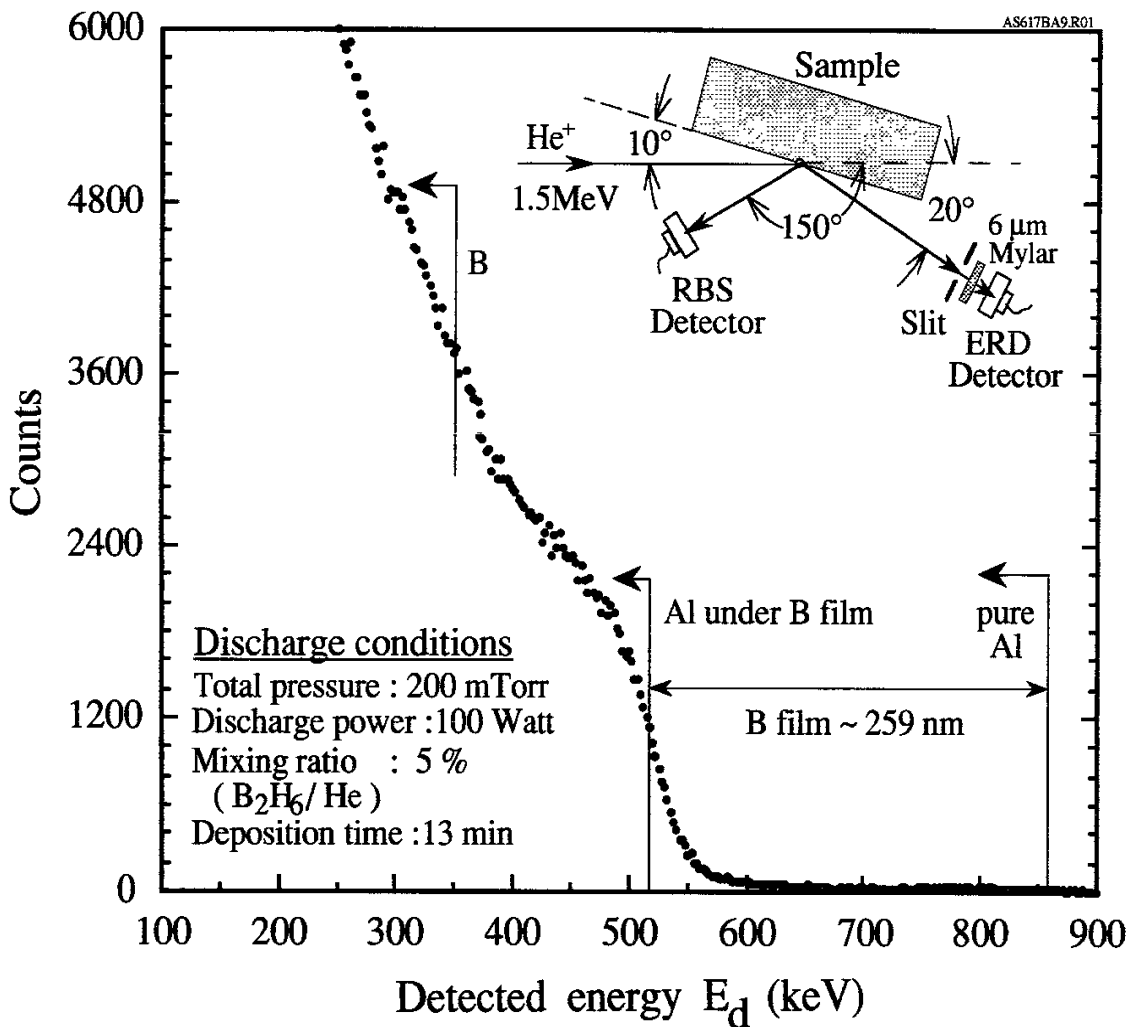


Figure 2, M. Natsir, et. al.

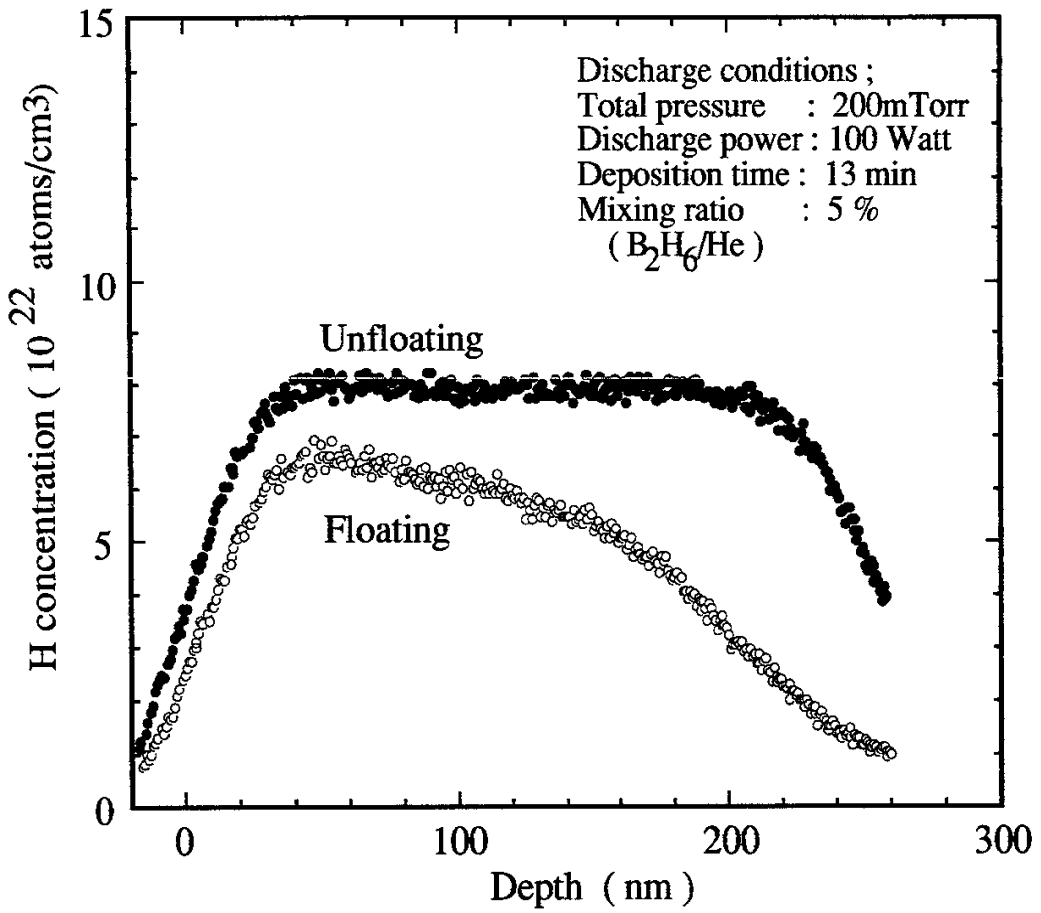


Fig. 3. M. Natsir, et. al.

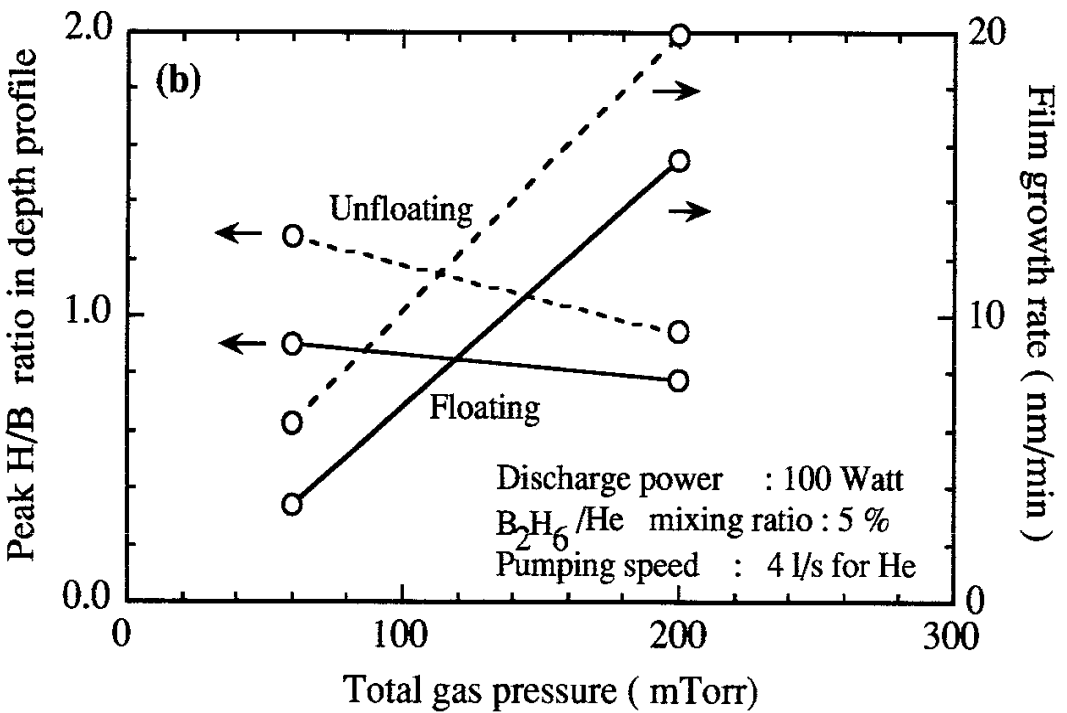
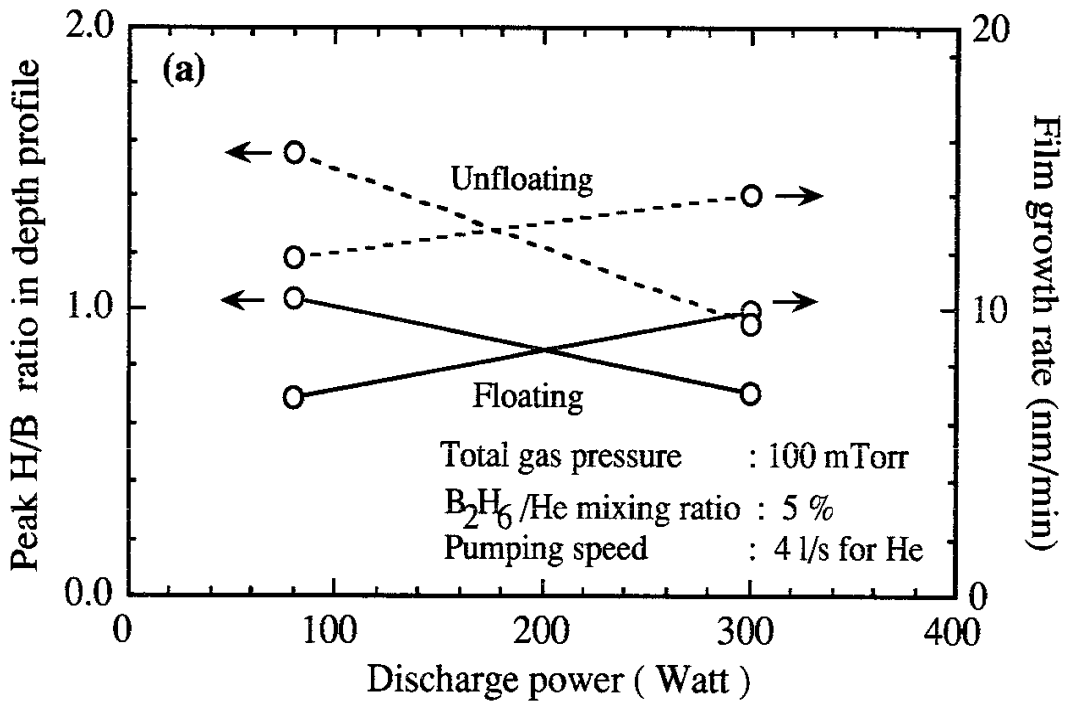


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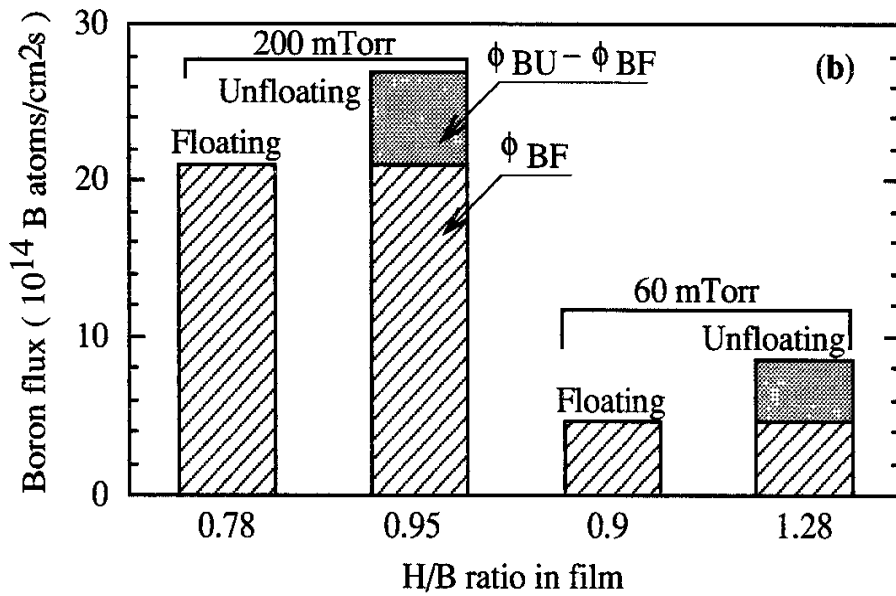
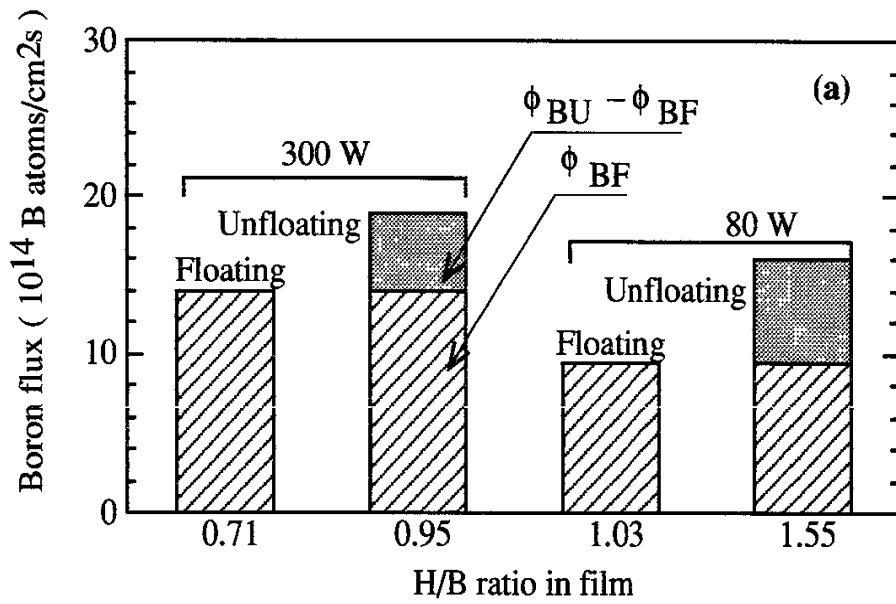


Figure 5, M. Natsir, et. al.

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