

## DEVELOPMENT OF THIN NCS-FOILS BY N<sup>+</sup> ION BEAM SPUTTERING AND THEIR CHARACTERISTICS

I. Sugai\*<sup>1)</sup>, M. Oyaizu<sup>1)</sup>, Y. Takeda<sup>1)</sup>, H. Kawakami<sup>1)</sup>, K. Kawasaki<sup>2)</sup>, T. Hattori<sup>2)</sup>

<sup>1)</sup>High Energy Accelerator Research Organization, Oho1-1, Tsukuba-shi, Ibaraki, 305-0801, Japan

<sup>2)</sup>Tokyo Institute of Technology, Ookayama, Meguro, Tokyo 152-8550, Japan

### Abstract

We have developed thin Nitride Carbon Stripper foils (NCS-foil) with a higher nitrogen content by ion beam sputtering method with reactive nitrogen gas. Such NCS-foils have been demonstrated that the foils in range of 10-25  $\mu\text{g}/\text{cm}^2$  have been shown to be long-lived as a stripper foil against high intensity heavy ion beam bombardment. From the results obtained by experimentally studies so far, we found that the nitrogen in the carbon foils plays very important role of the foil lifetime. Therefore, in order to investigate further influence of the lifetime on the nitrogen amount in the NCS-foils, we measured the sputtering yield at the different sputtering angles, fixing at 10 kV sputtering energy. The lifetime measurement of these foils was carried out with a 3.2 MeV Ne<sup>+</sup> ion beam. We also measured the thickness ratio of nitrogen to carbon in these foils by means of Rutherford Backward Scattering (RBS) method.

### INTRODUCTION

With great development of the beam intensity in heavy ion accelerators, the lifetime of carbon stripper foils becomes very shorter and thus such serious problems must be accepted in the operational efficiency of accelerators. In order to reduce this problem, long-lived carbon stripper foils of thickness of < 30  $\mu\text{g}/\text{cm}^2$  are still very important. Therefore, new type preparation methods have been developed world wide [1-4].

Among these methods, ion beam sputtering technique does not seem to be a suitable method because of its low sputtering yield and thus the method is used for small-scale production, compared with other well established methods [1-4]. However, in our previous works we have demonstrated that carbon foils made by heavy ion-beam sputtering (HIBS)[6], mixed ion-beam sputtering (MIBS)[7] and the ion beam sputtering with reactive nitrogen gas (IBSRN)[5,8-9] showed to be had the high potential for stripper foils under heavy ion irradiation of Ne<sup>+</sup> ion beam of 3.2 MeV energy. Among these, the foils made by the IBSRN method showed considerably long lifetime and mechanically strong compared with other tested foils including commercially available (CM) foil.

In order to know further the NCS-foils, we investigated following influence parameters on the foil lifetime; sputtering yields at different sputtering angles by N<sup>+</sup> ion beam sputtering, and the thickness ratio of nitrogen to

carbon in the each foils made at the sputtering conditions above mentioned.

### EXPERIMENTS

#### Foil preparation

The ion-beam sputtering system is almost the same as the one described in the previous paper [9]. An Al target holder equipped with four substrates of 27 mm x 36 mm was installed for collecting sputtered carbon material. These slides were previously coated with Crème-Cote releasing agent film of 2-4  $\mu\text{g}/\text{cm}^2$  (Crème-Cote, manufactured by J. Varley and Sons, St. Louis, Missouri, USA). These glass slides were placed on the target holder at 50 mm source to substrate distance. The aluminium target holder was placed at an angle of 45° relative to the primary ion beam direction.

The deposition was continuously monitored by a current integrator. The average thickness tested was 20±5  $\mu\text{g}/\text{cm}^2$ . The ion beam was stable and continuously regulated so as to fix the beam spot on the carbon target materials during sputtering. To thin foil < 10  $\mu\text{g}/\text{cm}^2$  could not always be floated from the glass substrates in distilled water, where thick foils > 10  $\mu\text{g}/\text{cm}^2$  could smoothly be floated from release agent Crème-Cote and mounted on 0.2 mm thick stainless steel target frame, these stripper foils were prepared at 10 kV sputtering voltage and 0.8-1.0 kV einzel lens voltage to focus the beam spot to 2-3 mm in diameter.

#### Measurement of sputtering yield

Sputtering yield at different sputtering angles at 10 kV for synthetic diamond (DM) and for glassy amorphous (GA) carbon sputter target materials has not been published so far as well as we know. Sputtering yield (atoms/ion) is expressed as simply the number of atoms of sputtered material per impinging ion. We used three different crystalline target materials of the DM, the GA and the poly-graphite (PG) carbon materials. The sputtering yield is dependent on; the mass of the incoming ion, the energy of the ion, the incident angle of the ion beam and the surface conditions of the sputter target material and its purity. Table 1 shows running parameters of the N<sup>+</sup> ion beam sputtering method. Figure 1 shows schematic of the sputtering layout. In order to know the sputtering yields for these crystalline carbon materials, we measured the sputtering yield versus sputtering angle ( $\alpha$ ) as shown in

\*Isao.sugai@kek.jp

Fig. 3 of Ref.[10] for the case of the IBSRN method. The sputtering yields were determined by the integrated beam current and the weight difference of the sputter material before and after sputtering. The surface of the target disc (15mm,  $\Phi$  and 3 mm height) was made to be as flat as possible. The target disc was fixed on the relevant seven beam stoppers made for 15°, 20°, 30°, 45°, 60°, 75° and 90° relative to the perpendicular ion beam. The layout for measuring of the sputtering yield was almost the same as shown in Fig. 1 of Ref.[10]. Sputtered carbon was collected on glass slides, which were mounted at a distance of 5 cm from the sputter source. In this experiment, we measured the sputtering yield at each inclination angle at least three times to reduce the distribution error of the obtained values. Figure 1 (a) and (b) shows the relation of the sputtering yield versus inclination angle ( $\alpha$ ) of the sputter source surface relative to the vertical ion beam for  $N^+$  ion beam. As usual the sputtering yields decreased with the sputtering angle ( $\alpha$ ). From figures, as usual, the yield values measured decreased with sputtering angle ( $\alpha$ ). Among these three tested target materials, the yield of the GA carbon showed highest yield of 1.3 than these 1.0 of the PG carbon at the sputtering angle of 15° and 1.0 of 20° for the DM material.

Table 1: Running parameters of the  $N^+$  ion beam sputtering method

#### Running parameters of the $N^+$ ion beam sputtering method

Ion species	$N_2$
Ion current	0.5-1.5 mA
Source gas purity	99.999%
Target materials	Poly graphite ;99.999%(Toyo Co. Japan) Man-made diamond (99.99%) Glassy amorphous carbon 99.99%
Vacuum	Vacuum gauge reading about $4 \times 10^{-4}$ Pa
Filament	Filament current 40-70 A (Ta, $\phi$ 1.5 mm)
Extraction voltage	5, 10 and 15 kV
Focus voltage	1.0-2.0 kV
Beam spot size	2-3 mm in diameter

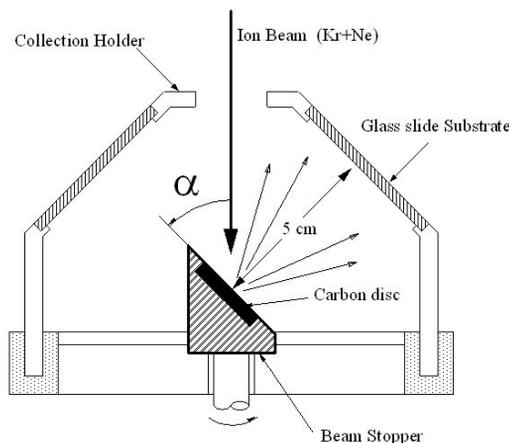


Figure 1: Schematic layout for measuring of sputtering yield.

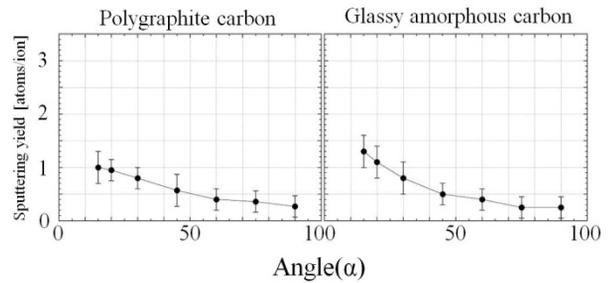


Figure 2: Relation of the sputtering yield versus inclination angle ( $\alpha$ ) in the RG-carbon (a) and the GA-carbon (b) targets.

#### Lifetime measurement

In order to investigate the sputtering angle ( $\alpha$ ) dependency on foil lifetime, we measured the lifetime of the carbon stripper foils made at seven sputtering angles ( $\alpha$ ) through 15° to 90°. The lifetime measurement of the foils was carried out with a 3.2 MeV  $Ne^+$  ion beam of  $3.0 \pm 0.5 \mu A$  and a beam spot of 3.5 mm  $\phi$  provided from Van de Graff accelerator at Tokyo Institute of Technology. Lifetime is defined as integrated ion current per unit area of  $cm^2$  until foil rupturing occurs. We measured at least three foil samples of each target material to reduce the distribution error. The error represents standard deviations.

The results are shown in Fig. 2 (a) and (b). As can be seen from the figures, the foil lifetimes indicated not so strongly dependent on the sputtering angle ( $\alpha$ ). Namely, the foil lifetimes in average for the PG-foil at the 15° showed 5250  $mC/cm^2$ , 6500  $mC/cm^2$  for 45° and 5250  $mC/cm^2$  for 90°. On the other hand, the lifetimes of the GA-foils showed 8000  $mC/cm^2$  for 15°, 5200  $mC/cm^2$  for 45° and 6000  $mC/cm^2$  for 90°. The characteristics presented of the sputtering angle by the  $N^+$  ion beam sputtering showed not great difference compared with the characteristics at the sputtering angle obtained by the (Kr+Ne) of MIBS method [10], which showed noticeably sputtering angle dependence.

Figure 3 (a) and (b) shows the relation of the foil lifetimes of the PG and GA-foils versus different sputtering angle ( $\alpha$ ). From the both pictures, we cannot see clearly difference sputtering angle ( $\alpha$ ). Among them, however, foils made at sputtering angle of 15° using the GA carbon target showed drastically long lifetime of 8000  $mC/cm^2$  in average, which corresponds to about 266 times that of the CM-best foils and a foil made with same conditions above showed 1200  $mC/cm^2$  in maximum, which corresponds to 400 times longer than that of the CM-foils.

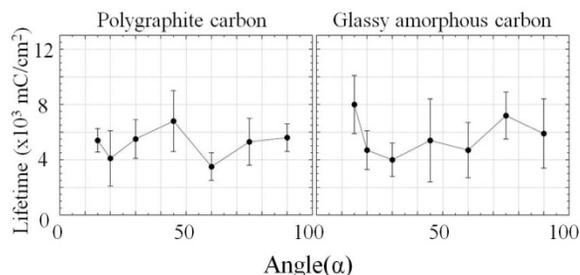


Figure 3: Relation of the sputtering angle versus foil lifetime in the PG and GA carbon target materials.

*Measurement of thickness ratio of nitrogen to carbon in nitride carbon foils*

In order to know the nitrogen content as a function of different sputtering angle ( $\alpha$ ), we measured the thickness ratio of nitrogen to carbon in these foils made by the seven different sputtering angles. The element analysis of nitride carbon foils was measured by the RBS using a 2MeV He<sup>2+</sup> beam provided from the Van de Graff accelerator at Kyoto University. The result showed in Figure 4 (a) the PG carbon foil and (b) the GA carbon foils. From both figures, we could not see that there are no clear dependence on the different sputtering angle as well as the result of thickness ratio of nitrogen to carbon in each foils made at different sputtering voltages could be clear not seen.

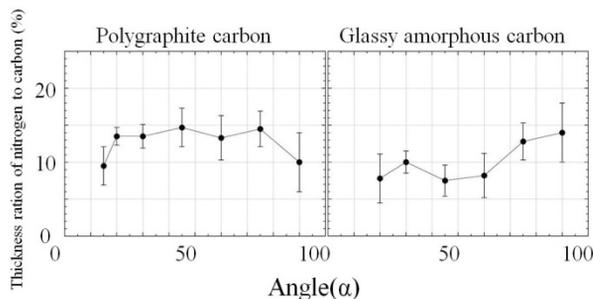


Figure 4: Thickness ratio of nitrogen to carbon in each foil made at different sputtering angle ( $\alpha$ ) using the PG and GA carbon target materials.

**SUMMARY**

We investigated influence parameters on the foil lifetime made by the IBSRN method: the sputtering yields at the different seven sputtering angles ( $\alpha$ ) through 15° to 90°, fixing at the sputtering voltage of 10 kV. The lifetime of these foils was measured with a 3.2 MeV Ne<sup>+</sup> ion beam.

The thickness ratios of the nitrogen to carbon in each foils made at the same sputtering conditions mentioned above were also measured with the RBS method. For these measurements, the DM, the PG carbon and the GA carbon materials were used. From the presented investigations, we found following characteristics;

1) the sputtering yield decreased with increasing of the sputtering angle ( $\alpha$ ) as usual, where the maximum and average yields in the GA carbon at sputtering angle of 15° showed rather high 1.8 and 1.3, which correspond to each

about 8 and 6.5 time higher than that of 0.2 at the normal sputtering angle of 90°. Hence, the foil preparation time could be reduced to about 1/9 or 1/6 compared to the normal sputtering angle of 90° so far.

2) The lifetime of the foils does not noticeably depend on the sputtering angles like the MIBS method [10],

3) Thickness ratio in each foils made at different sputtering angle did not noticeably depend on the sputtering angle ( $\alpha$ ). However, several foils made at the 15° and when using the GA carbon material showed drastically long lifetime of higher than 8000 mC/cm<sup>2</sup>, which corresponds to about 266 times compared to the CM best foils.

In conclusion, We turned out that effective stripper foil preparation with drastically long-lived >8000 mC/cm<sup>2</sup> could be prepared by using the sputtering angle of 15° and the GA carbon target material using N<sup>+</sup> ion beam sputtering.

**REFERENCES**

- [1] N. R. S. Tait, B. H. Armitage, D. S. Whitmell, Nucl. Instr. and Meth. 167 (1979)21
- [2] G. Dollinger, P. Maier-Komor, Nucl. Instr. and Meth. B 53 (1991) 352
- [3] R.L. Auble, J.K. Baier, D.M. Galbraith, C.M. Jones, P.H. Stelson, D.C. Weisser, Nucl. Instr. and Meth. 177 (1980)289
- [4] I. Sugai, T. Hattori, H. Suzuki, and H. Kinoshita, H.Kato, K.Yamazaki, Nucl. Instr. and Meth. A267(1988)376.
- [5] I. Sugai, M. Oyaizu, T. Hattori, K. Kawasaki, T. Yano, H. Muto, Y. Takahashi, K.Yamazaki, Nucl. Instr. and Meth. A 303 (1991) 59.
- [6] I. Sugai, Y. Takeda, M.Oyaizu, H. Kawakami, Y. Hattori, K. Kawasaki, T. Yano, H. Muto, Y. Takahashi, Y. Ishii, F. Hirata, M. Okamura, Nucl. Instr. and Meth. A320 (1992) 15.
- [7] H. Muto, M. Oyaizu, I. Sugai, K. Kawasaki, Y. Takahashi, Y. Ishii, T. Hirata, T. Hattori, Nucl. Instr. and Meth. B83 (1993) 291
- [8] I. Sugai, M. Oyaizu, H. Kawakami, T. Hattori, H. Tomizawa, K. Kawasaki, Nucl. Instr. and Meth. A 397 (1997) 137.
- [9] I. Sugai, Y. Takeda, M.Oyaizu, H. Kawakami, Y. Hattori, K. Kawasaki, Nucl. Instr. and Meth. A 590 (2008) 37.
- [10] I. Sugai, M.Oyaizu, Y. Takeda H. Kawakami, Y. Hattori, K. Kawasaki, Nucl. Instr. and Meth. A 617(2010) 448